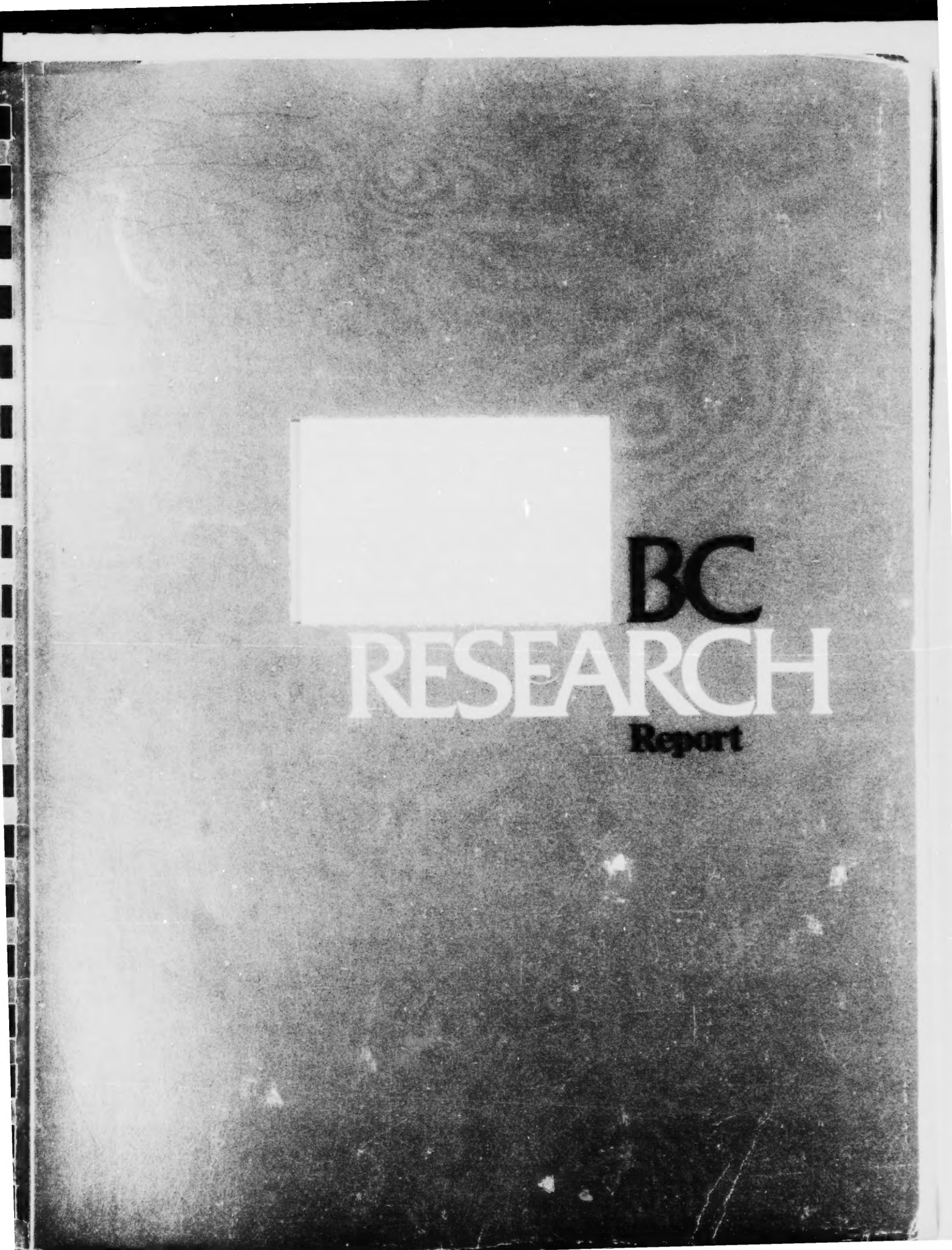

BC
RESEARCH
Report

4-9081

CONTRACT REPORT NO. OSQ84-00139

PROJECT REPORT

CONTINUATION OF THE EVALUATION OF HAT CREEK COAL FOR DIRECT LIQUEFACTION

Prepared for:
SERIAL # OSQ84-00139
Canada Centre for Mineral and Energy Technology
(DSS File 23SQ-~~4-9681~~)
4-9081

B.C. Hydro and Power Authority Ltd.

and

B.C. Ministry of Energy, Mines and Petroleum Resources

Prepared by:

Chemical Technology Division
B.C. Research
3650 Wesbrook Mall
Vancouver, B.C.
V6S 2L2

MICROMEDIA

December, 1985

104 pp

TABLE OF CONTENTS

	Page
LIST OF TABLES	
LIST OF FIGURES	
LIST OF APPENDICES	
1.0 SUMMARY	1
2.0 INTRODUCTION	4
2.1 Background	4
2.2 Objectives	5
3.0 MATERIALS AND METHODS	6
3.1 Test Coals	6
3.2 Liquefaction Solvent	7
3.3 Reagents	7
3.4 Tubing Bomb System	7
3.5 Continuous Liquefaction System	8
3.6 Continuous Hydrotreater	9
3.7 Analytical Procedures	9
3.7.1 Slurry Feed and Products	9
3.7.2 Gas Analyses	10
3.7.3 Elemental Analyses	11
3.8 Conversion Calculations	11
4.0 RESULTS AND DISCUSSION	12
4.1 Test Coals	12
4.2 Systems Performance	13
4.2.1 Tubing Bomb System	13
4.2.2 Continuous Hydrotreater	14
4.2.3 Continuous Liquefaction System	15

TABLE OF CONTENTS (Cont'd)

		Page
4.3	Continuous Hydrotreating	15
	4.3.1 Hydrogenation of Creosote	15
	4.3.2 Hydrotreating of Liquefaction Products	19
	4.3.3 Gas Production from Continuous Hydrotreating	20
	4.3.4 Hydrogen Consumption in Continuous Hydrotreating	20
4.4	Continuous Liquefaction	21
	4.4.1 Hydrogen Availability from Reforming C_1-C_3 Hydrocarbon Byproduct Gas	22
4.5	Product Characterization and Potential Utilization	23
	4.5.1 Net Product	23
	4.5.2 Net Product Properties and Utilization	24
	4.5.3 Distillation and Coking Behaviour of Product Liquid and "Sludge"	26
4.6	Chemical Stability of Tetrahydrofuran	28
4.7	Solid Waste Disposal	28
4.8	Economic Analysis	30
	4.8.1 Capital Costs	32
	4.8.2 Manufacturing Cost	33
	4.8.2.1 Coal	33
	4.8.2.2 Electric Power	34
	4.8.2.3 Catalyst and Royalty	34
	4.8.2.4 Labour	36
	4.8.3 Revenue	36
	4.8.3.1 Rate of Return and Payback	37
	4.8.4 General Evaluation	38

TABLE OF CONTENTS (Cont'd)

	Page
5.0 CONCLUSIONS	42
5.1 Technical Results.	42
5.2 Economic Assessment.	43
5.3 Environmental Assessment	44
6.0 RECOMMENDATIONS	45
6.1 Liquid-Residue Separation	45
6.2 Coal Beneficiation	45
6.3 Co-processing	46
6.4 Binder Pitch Manufacture	46
6.5 Pilot and Demonstration Plant Testing	46

LIST OF TABLES

Table		Page
1	Proximate Analyses and Calorific Values Test Coals	50
2	Projected Run of Mine Hat Creek Coal Quality	51
3	Iron Contents of Test Coals Before and After Ion Exchange	52
4	Hydrotreater Test Data on Raw Creosote	53
5	Trends in Total Conversion with FeXC Catalyst and Increasing Solvent Donor Content	54
6	Hydrotreater Test Data on Retreatment of Hydrotreated Creosote, +200°C Fraction	55
7	Comparison of Products from First and Second Stage Creosote Hydrotreating	56
8	Hydrotreater Test Data on Continuous Liquefaction Products	57
9	Gas Production Hydrogenation of Coal Liquefaction Products	58
10	Ultimate Analysis, Atomic H/C Ratios and Composition Changes of Selected Hydrotreated Feeds and Products	59
11	Hydrotreater Hydrogen Consumption	60
12	Summary of Continuous Liquefaction Test Runs on Hat Creek Coals	61
13	Calorific and Gravimetric Conversions from Continuous Liquefaction	62
14	Comparison of Gas Production in Tubing Bomb	63
15	Partial Chromatographic Analyses of Naphtha Before/ After Aqueous Acid/Alkali Stripping	64
16	Elemental Analyses of H31 Naphtha Before/After Aqueous Stripping	65

LIST OF TABLES (Cont'd.)

Table		Page
17	Results of Sealed Tube	66
18	Comparison of Unit Operations for Indirect vs. Direct Liquefaction	67
19	Comparison of Cost and Revenue Estimates for Direct Liquefaction of Hat Creek Coal	68

LIST OF FIGURES

Figure		
1	Schematic Diagram of Tubing Bomb Reactor	69
2	Continuous Coal Liquefaction Unit	70
3	Continuous Hydrotreater	71
4	Thermogravimetric Analysis of C37 Primary Liquid Product	72
5	Thermogravimetric Analysis of C37 Slude Product . . .	73
6	Thermogravimetric Analysis of C37 Product (+190°C) After Hydrotreating	74
7	Thermogravimetric Analysis of C37 Residue	75

LIST OF APPENDICES

Appendix	
1	Summaries of Hydrotreater Operational Test Data
2	Summaries of Continuous Liquefaction Operational Test Data

1.0 SUMMARY

In fiscal 1984, B.C. Research performed continuing studies on direct liquefaction of Hat Creek coal under a cost sharing agreement involving CANMET, B.C. Hydro and the B.C. Ministry of Energy, Mines and Petroleum Resources.

Primary objectives of the current program include detailed mass balances and product slate evaluation for both the direct liquefaction and product hydrotreating operations. Secondary objectives include optimization and end use compatibility of net liquid product, assessment of separation solvent stability and waste disposal options.

Completed testwork has included modification to both continuous operating systems to allow quantitative product recovery.

Mass balances for the hydrotreating operation of 100±1% have been obtained with corresponding data on relative yields of gas (C_1 - C_4) hydrocarbons, naphtha (C_5 - 190°C) product and recycle solvent.

The initial set of modifications to the continuous liquefaction system gave acceptable slurry product recovery but did not give acceptable data on C_1 - C_4 gas products. A subsequent modification of the sampling procedure has given data indicating acceptable overall material balances of 97-100% for runs with 89-92% gravimetric total conversion.

Current testwork confirms the useful effect of iron catalyst on conversion as well as on mineral matter rejection from raw coal.

Hydrotreating test data on liquefaction products confirm previous data indicating that the gross product comprises hydrocarbon gases (C_1-C_4) and liquids boiling below ~190°C. All remaining liquid is recycled as process donor solvent.

Extended continuous operation of the upflow, fixed bed solvent hydrotreater system (199 kg feed/kg catalyst) was obtained without measurable loss of activity or operational problems such as coking. Commercially available ebulliated bed hydrotreating would then be suitable for large scale operation.

Conservative estimation of process hydrogen requirement at 3-3.7 g/100 g daf indicates that all C_1-C_4 byproduct gases could be consumed in steam reforming to make the process self sufficient - i.e. no makeup natural gas. On this basis, overall net calorific conversion efficiency is projected to be ≈63% to a C_5-190°C naphtha containing ~3% of phenolics separable by aqueous caustic stripping. Nitrogen and sulphur levels in aqueous acid/alkali stripped naphtha were <0.05% ppm and <0.02% ppm respectively.

Test data indicate that tetrahydrofuran (THF) is unstable to hydrolysis during steam stripping in the presence of liquefaction residue; future testwork should include evaluation of developing liquid-residue separation technologies potentially capable of achieving recoveries equivalent to those attainable with THF.

Aqueous leachate from liquefaction residue is toxic to fish, so landfill disposal will require use of sealed impoundments.

Economic analysis indicates that direct liquefaction of Hat Creek coal could be economically viable at current liquid fuel prices if a subsidy in the form of a gasoline tax remission were available to help defray capital costs. Comparative analysis suggests direct liquefaction of Hat Creek coal is attractive in comparison to some frontier and heavy oil-bitumen developments. Advantages include existing transport and social infrastructure, large single site reserves and low mining costs.

Recommendations for further study include investigation of advanced coal beneficiation methods (oil agglomeration) for ash reduction and investigation of potential direct marketing of the primary "sludge" (i.e. material insoluble in ambient temperature process solvent) as electrode binder pitch.

2.0 INTRODUCTION

2.1 Background

B.C. Research has had an active program on direct coal liquefaction since 1979. The first phase of this program, supported by the government of British Columbia, involved bench scale testwork on samples of all economically accessible British Columbia coals. These tests were designed to identify coal types and/or specific coals having good reactivity - i.e., high conversion to light liquid products - and hence being potential feedstocks for commercial direct liquefaction. Results of this program indicated that sub-bituminous coals from the Hat Creek deposit can give attractively high conversions ($\geq$90%) to liquid and gas under realistic process conditions.

Further testwork up to July, 1984 confirmed that the high conversions observed in batch tests could be duplicated in a continuous bench scale apparatus treating 1-2 kg/h of coal-solvent slurry. Results of that work also indicated that a simple pretreatment of coal with aqueous ferrous sulphate results in reduced coal ash content and useful catalysis of liquefaction reactions by iron incorporated into the coal matrix during the aqueous "ion exchange" procedure. From those results it was concluded that further work on the continuous bench scale system was required to generate more detailed information on critical process areas including mass balances, product-residue separation, product quality and environmental stability of wastes (1). Joint funding of this program by CANMET, B.C. Hydro and the B.C. Ministry of Energy, Mines and Petroleum Resources allowed completion of this current program.

2.2 Objectives

With reference to the project proposal (2), technical objectives of the work covered in this report include:

- generation of mass and chemical balances for the three critical process areas: liquefaction, product-residue separation and product hydrotreating.

- determination of the chemical stability of tetrahydrofuran under conditions required for use in liquid residue separation.

- optimization of net liquid product in terms of material removed from the liquefaction/hydrotreating/solvent recycle system and its further chemical treatment.

- determination of net product compatibility with existing market fuel specifications.

- evaluation of options for disposal of solid and liquid process wastes.

- overall economic assessment of direct liquefaction of Hat Creek coal with reference to current and future markets for liquid and gas products.

3.0 MATERIALS AND METHODS

3.1 Test Coals

Coal samples for testing were obtained by channel sampling of bulk coal stockpiles at the minesite. These stockpiles were established by B.C. Hydro and Power Authority Ltd. (leaseholder for the deposit) as part of a major sampling effort. The stockpiles, designated "Trench A" and "Trench B" were taken from large exploration pits; they represent major zones of the proven, mineable reserves of the deposit.

Samples were bagged and labelled at the site. In the laboratory, Trench A and Trench B samples were separately bulk blended, air dried, crushed through jaw and gyratory crushers, riffle split and rebagged in ~10 kg subsamples for storage.

As required for testwork, subsamples were recovered from storage, impact milled in a CO_2 atmosphere to -150 microns (-100 mesh) and subsampled for analysis.

For non-catalyzed liquefaction tests, -150 micron material was slurried with solvent and passed through a vibratory ball mill for final comminution.

For tests involving iron catalyst added by ion exchange (3) the following procedure was evolved:

- slurry 40 kg coal (-150 micron) with 200 L tapwater and 15 kg technical $FeSO_4.4H_2O$
- agitate for 48 h and settle overnight
- decant solution and non-settling fines to separate vessel
- repulp with 3 x 100 L tapwater, settle and decant

- transfer settled ("coarse") solids to tray and dry at 40°C in air
- pass dried material through impact mill under CO_2 to break up agglomerates
- slurry with solvent and proceed to vibration milling

3.2 Liquefaction Solvent

Commercial creosote oil purchased from Crawley Tar Ltd. was the starting material for all tests. This raw material was prepared by hydrotreating and distillation as described in the following sections. Where available, liquefaction products from previously reported (1) testwork were used in blends with hydrotreated creosote oil.

3.3 Reagents

Reagents used in sample processing were tetrahydrofuran, carbon disulphide and toluene; technical grades were used except as specifically noted.

Reagent grade chemicals were used in preparation of standard mixtures for calibration of the capillary gas chromatograph used in analysis of liquid samples.

3.4 Tubing Bomb System

The "tubing bomb" microautoclave batch test facility is shown schematically in Figure 1. The system and method of operation have been described previously (4).

3.5 Continuous Liquefaction System

The bench scale liquefaction system is shown schematically as Figure 2. The feed system is of conventional design. It consists of an agitated slurry feed tank (load cell mounted), a Moyno pump to provide feed circulation and a two-head Bran & Lubbe high pressure piston pump which delivers slurry to the reactor. The reactor is a 2 L Autoclave Engineers autoclave with a magnedrive unit driving a 2.5 cm diameter, four blade turbine.

A single, large capacity (32 L) receiver was installed in place of two sets of primary/secondary receivers used in previously reported work. This arrangement eliminated the necessity of switching between the receivers as they filled with product. The bulk receiver (gas cylinder) is mounted in an inverted position to allow clean gravity drainage of the product slurry. This facilitates quantitative product slurry recovery as required for material balance tests.

As shown in Figure 2, a back pressure regulator controls gas outflow from the system through a rotameter, sampling port and vent.

Hydrogen feed to the liquefaction system (and the hydrotreater) was via a pressure regulator and hydrogen flow controller as shown in Figure 2.

Operating and product workup procedures were as previously reported (1).

3.6 Continuous Hydrotreater

The fixed bed continuous hydrotreater used in the study is shown schematically as Figure 3. The load cell mounted feed tank is located above the high pressure Bran & Lubbe feed pump to provide inlet pressure. Pressurized liquid feed passes through a preheater and joins the hydrogen feed at the base of the reactor column. The reactor (5 cm diameter x 2 m, 316 stainless steel tube) is heated by three independently controlled electrical clamshell heaters. Reactor outflow (liquid, vapour and gas) passes to a 20 L receiver vessel (gas cylinder) for cooling and phase separation. Outflow gas, controlled by a back pressure regulator, passes via a sampling port and rotameter to an exterior vent.

Hydrogen flow to the unit was controlled by a Matheson SP1731 Mass Flow Controller.

Catalyst in the reactor was not changed during the test program; the catalyst and presulphiding procedures were reported previously (1).

3.7 Analytical Procedures

3.7.1 Slurry Feed and Products

For tubing bomb tests, product slurry was washed out with tetrahydrofuran and filtered on tared glass fibre (0.45μ) pads. Filter cakes were washed with THF at ambient temperature, vacuum dried and weighed to determine solid residue. THF soluble product was diluted to a fixed volume (normally 100 mL) with THF; aliquot samples of this solution were further diluted with carbon disulphide before capillary gas chromatography using a Hewlett Packard instrument with a 15 m capillary column and flame ionization detector.

For thermogravimetric analyses (TGA) using a Dupont 1080 unit, aliquot samples of THF solubles were evaporated under vacuum at ~40°C; samples of remaining liquid were transferred to the TGA balance pan using a syringe. TGA analyses of THF extract and hydrotreater liquid product were done under flowing nitrogen with programmed temperature increase of 20°C/minute to 300°C for estimation of true distillables or to 525°C for estimation of carbon or coke residue. Proximate analyses of THF insolubles were done by TGA after manual removal of the filter cake from the pad and mixing. Calorific values were determined for separate subsamples of residue using a Parr calorimeter.

Slurry product from the continuous reactor was analyzed in a similar manner, except that weighed aliquot samples were used.

Reactor feed slurry was analyzed by THF filtration of weighed samples to confirm feed solids content.

3.7.2 Gas Analyses

Gas samples from all reactors were taken at atmospheric pressure in SKF sample bags. From the bag, a subsample was transferred to the chromatograph injection loop by connecting the sample bag port to an injection tube followed by flushing and filling the injection loop using manual pressure on the bag.

For the continuous reactors, multiple samples were required during both the operating period and depressurization of the receivers in order to establish total gas production. For tubing bombs, a single bag sample was obtained.

Gases measured directly by gas chromatography (GC) were N_2, O_2, CO, CO_2 and C_1-C_6 hydrocarbons; hydrogen was normally measured by difference.

3.7.3 Elemental Analyses

Elemental (CHNOS) analyses were subcontracted to Canadian Microanalytical Ltd.

3.8 Conversion Calculations

Conversion data are presented for both gravimetric and calorific conversion of coal. For samples from the continuous system, both methods rely on (TGA determined) ash content of insoluble residue. For tubing bomb tests, the total residue weight is used.

4.0 RESULTS AND DISCUSSION

4.1 Test Coals

Proximate analyses and calorific values for the Trench A and B coal samples used are presented in Table 1. For comparison purposes, Table 2 presents anticipated run of mine coal quality data prepared by B.C. Hydro (5) for selective mining to support a thermal power plant consuming 9.7×10^6 tonnes/year over a 20 year operating life.

Data of Tables 1 and 2 clearly indicate the high ash and moisture contents of run of mine coal. However, selective mining as planned for a thermal power plant results in run of mine coal at ~33.5% ash (dry basis) compared to 47% and 35% for Trench A and B respectively. Some ash reduction is obtained by rejection of fine clay during the iron ion exchange procedure, so if the anticipated production blend were used, ash content of liquefaction feed would be in the 28-31% range (dry basis) or roughly as tested for the Trench A and B samples.

There is a significant difference between the projected dry coal calorific value of 18.06 MJ/kg and the (calculated) dry coal calorific values of 16.5 and 17.1 MJ/kg for ion exchanged Trench A and B samples respectively. The discrepancy on an ash free basis would be even larger, since both ion exchanged samples are lower in (dry basis) ash contents than the projected run of mine coal.

Whether the discrepancy arises from aging effects or a better quality of coal, the higher calorific value of projected run of mine coal would obviously be advantageous for liquefaction at equivalent conversion levels.

Another significant difference between projected coal quality and test material is iron content. Table 3 shows dry basis iron contents of test coals before and after the ion exchange procedure. The projected

run of mine coal contains 2% iron (2.8% Fe_2O_3) which is significantly higher than either test coal. Thus, if the incremental iron is in a form which can be activated, the amount of iron required to obtain test-indicated catalytic effects (~1%, see Table 3) may be less than required for the test samples.

The ratio of volatile matter to fixed carbon is also significantly different for the projected run of mine material (0.98) as compared to values for Trench A and B test coals (1.13 and 1.26 respectively for ion exchanged samples). The bound water contents of clay and other mineral constituents of Hat Creek coals are known to contribute spuriously to volatile matter, and this may explain part of the difference. Other potential causes include slight differences in rank, analytical methods, and (possibly) atmospheric oxidation effects due to long term open storage of test coals.

4.2 Systems Performance

The following sub-sections provide a critical overview of performance of the test systems as a basis for assessment of test data.

4.2.1 Tubing Bomb System

Tubing bomb tests were done in parallel to most of the continuous liquefaction tests using samples of feed slurry, as well as to determine conversion trends with solvent quality (tetralin:naphthalene or T:N ratio), solvent:coal ratio, and catalyst components. Reproduceability of results in this system is generally very good with respect to conversion, gas production and hydrogen consumption. Data reported previously (1) and in following sections of this report confirm that total conversions achieved in tubing bomb tests can be duplicated in the continuous system. However, the accurate control of residence time in batch tubing bomb tests allows accurate evaluation of gas production vs. time whereas the short-circuiting necessarily associated with a single stage, back mixed system only allows measurement of an average residence time.

Considering that virtually all schemes for commercial scale liquefaction involve multi-stage series reactors to control residence time, the capability of the tubing system to model a closely controlled residence time is clearly an advantage in relating results to a large scale operation.

4.2.2 Continuous Hydrotreater

Operation of the fixed bed hydrotreater in an upflow mode has proven to allow long term stable operation without any observed tendency to coking (at least on a macroscopic scale) or catalyst deactivation. The system functions reliably for production of kilogram quantities of hydrotreated products, temperature control is good, and a considerable range of pressure-temperature-flowrate conditions can be used.

Limitations of the system arise mainly from its size (with a consequent requirement for large volumes of feed), and the unresolved problem of obtaining reliable on-line samples. Several approaches to on-line product sampling were attempted during the current program; none of the methods were successful in providing a representative liquid-gas sample. Consequently, reported results are time averaged for the operating period plus startup and shutdown. Gas products are determined by sampling/analysis of exhaust gas during the operating period and during depressurization of the entire system including the reactor column. Thus, although operational control of the unit is more than sufficient to obtain multiple steady states within a single run, sampling requirements dictate that only one steady state can be tested per run. Even within this restriction, there appears to be significant variation and/or systematic errors in critical parameters including gas production and - particularly- hydrogen consumption as indicated by gas analyses. Overall mass balances and liquid recovery are considered acceptable.

4.2.3 Continuous Liquefaction System

Operation of the continuous liquefaction system is fully satisfactory with regard to temperature control, gas and liquid feedrates. Limitations of the system are due to its single-stage, back-mixed reactor (i.e. control over average residence time only) and - as for the hydrotreater - lack of a workable system for equilibrium, on-line product sampling. The lack of workable on-line sampling is less of a constraint for the liquefaction system than for the hydrotreater, since optimum liquefaction conditions are fairly well defined. However, as for hydrotreating, determination of gas phase products requires multiple analyses, summing of the data and venting of the entire system to atmospheric pressure.

4.3 Continuous Hydrotreating

4.3.1 Hydrogenation of Creosote

Summarized results of continuous hydrotreating tests on creosote are presented in Table 4; data on individual runs are presented in Appendix 1.

The first sequence of runs (H17-H24, inclusive) was done to generate a stock of hydrotreated, distilled solvent for use as startup solvent in liquefaction tests, and also to establish methodology for material balances. A range of temperature and flow conditions was used to obtain performance data.

With respect to liquid product recovery (Table 4), it was found that holdup of liquid in the reactor column was inconsistent; recoveries varied in the range of 97-105% of liquid feed. Thus, in subsequent runs material remaining in the reactor was allowed to settle overnight (after cooling to T<200°C with hydrogen purging), drained out and added to liquid recovered from the receiver. This method added to the

complexity of operation, since it involved depressurization of the reactor as well as the receiver after each test run.

Product donor content, indicated by tetralin:naphthalene (T:N) ratio in the product, is a critical parameter in hydrotreater performance. As discussed below, products of H17-H24 were found to be inadequate with respect to donor content and had to be reprocessed. However, the results of variations in conditions are useful in estimating optimum conditions.

Considering the data of Table 4, for runs H17 and H18, the major difference is in temperature (310 vs. 325°C); the liquid flowrates were as close as could be obtained. The product T:N is lower for H18 suggesting that - under otherwise equivalent conditions - increased temperature does not favour donor formation. Run H19 with a low feedrate and increased hydrogen flowrate (5 L/min vs. 2.5 L/min in H17 and H18), gave the best T:N (3.4) of the entire test sequence. From the amount of liquid recovered by draining the reactor (~0.8 L) even after gas flushing, it is virtually certain that there is a continuous liquid phase present in the hydrotreater during steady state operation. Thus, the physical mixing and hydrogen transfer to solution due to the doubled hydrogen flowrate in H19 compared to H17 and H18 seems more likely to be responsible for the substantial increase in T:N ratio (3.4 in H19 compared to 0.67 in H18) than the minor (13.8 vs. 10.2) increase in hydrogen overpressure. This seems to be confirmed by the low product T:N of 0.85 in H20 in which the system pressure was maintained at 13.8 MPa but hydrogen flowrate was 2.5 L/min.

Results of H21-H23, all nominally at the same condition, are difficult to interpret. The large variations in T:N ratio do not seem to correspond to any measured parameter. One possibility is that variations in distillation procedure resulted in removal of tetralin (bp 208°C) in an inconsistent way.

Results of H24 are compatible with this interpretation since the product was distilled by both the single plate and reflux procedures.

At this point in the test program, a continuous liquefaction run (C33) was undertaken with solvent prepared by blending +200°C product from H12-H24 with T:N =1.0. Results (see below, Section 4.4) were unsatisfactory, so a series of tubing bomb tests was undertaken with a variety of coal and solvent samples to determine solvent performance vs. donor content. Results are presented in Table 5.

To isolate solvent donor content effects, Trench A and B coals were run with solvents having T:N ratios of 4.5 and 7.5.

Data from Table 5 indicate the effect of solvent T:N on total conversion as follows:

	Total Conversion		
	Solvent Tetralin:Naphthalene (T:N)		
	T:N=1	T:N=4.5	T:N=7.5
Trench A	81.3	82.0	84.5
Trench B	77.2,79.5	85.1	88.6,89.0

Results presented in Table 5 also show the progressive increase in conversion attainable by combining use of high donor content solvent with iron exchange and carbon disulphide. From these results it is reasonably clear that to obtain maximum total conversion within minimum reactor volume requires high solvent donor content and use of the iron exchange catalysis (Fe XC) procedure.

On the basis of these data, combined distilled (+200°C) product from runs H17-H24 was reprocessed through the hydrotreater. Data for these runs (H25 and H26) are presented in Table 6. Combined product from these runs totalled 100 ± 0.5% of liquid feed after full drainage of the reactor. The combined product from these runs (T:N = 3) was used in liquefaction tests as described below.

Major component analyses of liquid products from the primary (H24) and secondary (H25) creosote hydrotreating are presented in Table 7.

The final hydrotreater test on creosote (H32, Table 4) was done to determine the effect on catalyst activity of runs H27-H31 in which liquefaction products were used. Conditions were the same (within limits of experimental control) as for runs H21-H23.

Evaluation of any change in catalyst activity is complicated by the fact that in H21-H23 the distillation conditions were not closely controlled so there was some loss of tetralin to the light fraction. However the T:N ratio of 4.08 observed in H32 suggests that there was no significant deterioration in catalyst activity due to treatment of the liquefaction products.

After H32, the total age of the hydrotreater catalyst was 199 (kg liquid feed/kg catalyst); product T:N ratio is well within the range of values obtained with fresh catalyst (1) indicating no appreciable deterioration in catalyst activity. This suggests that catalyst activity under the conditions used reaches a plateau value quite early in its service life and that deterioration is at least slow in the range of exposure so far tested. The data are too limited to make any useful prediction of service life which would be thousands of kg/kg in equivalent operation on petroleum and would have to be of the same order of magnitude to be practical for coal liquefaction applications.

4.3.2 Hydrotreating of Liquefaction Products

Results of hydrotreater runs on liquefaction products are summarized in Table 8; elemental analyses of selected materials are presented in Table 9.

For the two tests - H27 and H29 - on Trench B coal liquefaction product, liquid recoveries were good (100%, 102%) in both tests.

Light (-180°C) liquid recoveries were significantly different (14% and 9%) respectively, while the carbon and hydrogen changes (feed to product, see Table 9) are so different that the analyses must be considered questionable.

Chromatographic analysis of products indicates similar contents of major components:

	H27 (Product)	H 29 (Product)
BTX (%)	1.33	1.17
Decalins (%)	4.53	3.80
Tetralin & Naphthalene	18.11	18.77

which suggests a close approach to equilibrium recycle conditions. For higher aromatics, conversions of phenanthrene and pyrene were consistent in both runs.

It is interesting to note that 9,10 dihydroanthracene is present in measurable concentrations (0.44, 0.40%) in the liquefaction product (ie donor depleted liquid) as well as in hydrotreated product (0.90 and 0.86%).

The apparent trend toward a lower weight fraction of light product (in this case boiling below 180°C) is potentially a concern.

In this regard the data of Table 7, which compares light liquid content and analyses of products from first and second stage hydrogenation of creosote oil, indicate that extra light liquid products can be generated of analogous composition (with somewhat higher indan and decalin content) by reprocessing previously hydrotreated material. The analogy is not exact, since the data refer to two stage treatment of creosote oil rather than liquefaction product. However the bulk compositions of the two materials are sufficiently similar to indicate analogous behaviour.

4.3.3 Gas Production from Continuous Hydrotreating

As previously discussed, the experimental system used places some limitation on the accuracy of data on gas phase (C_1 - C_4) products from the continuous hydrotreater. With this reservation, gas product data are presented in Table 9; amounts of hydrocarbon byproducts are low in all cases relative to yields from liquefaction.

4.3.4 Hydrogen Consumption in Continuous Hydrotreating

For reasons previously noted, directly measured hydrogen consumption in the continuous hydrotreater unit are not reliable. Therefore, hydrogen consumption values have been computed from analytical hydrogen contents of the feed, liquid and C_1 - C_4 gas products. Computed data are presented in Table 11.

The tabulated data clearly show that the hydrotreating operation is a substantial consumer of hydrogen. This is as expected, since the operation combines the functions of product upgrading - ie. hydrotreating to produce the bulk of the light distillate product, partial heteroatom removal, and regeneration of donor solvent to provide the bulk of hydrogen actually consumed in the liquefaction stage.

Although the C_1 - C_4 gas product of hydrotreating is low compared to the C_1 - C_4 product in liquefaction, the amount of hydrogen accounted for by these products is significant, as indicated by the data of Table 12.

Liquid recovery (vs. feed, w/w) for hydrotreating was uniformly high; specific gravities of liquid feed and products were not determined, but on the basis of reduced aromaticity, it is reasonable to expect that volume recoveries of liquid were in excess of 100%.

4.4 Continuous Liquefaction

Results of continuous liquefaction tests are summarized in Table 12.

For runs C34-37, all using iron exchanged coal, gravimetric total conversion are in the range of 89-92%, which is consistent with results of tubing bomb tests under equivalent conditions. Conversions by calorific value (Table 13) are somewhat higher, which is consistent with previous observations.

"Sludge" yields (THF soluble material, see Table 12) are consistently in the 10-12% range.

Overall material balances (Table 12) are consistently in the 97-99% range which is considered to be the limit of accuracy attainable with the gas sampling/analysis system used.

Hydrogen consumptions from the gas phase were not computed due to the (relatively) low accuracy of gas analyses.

Data on gas phase products for runs C34-C37 and parallel tubing bomb tests on feed slurries are presented in Table 17.

Carbon dioxide is the largest indicated component (8.3-12.8 wt% vs. daf coal); variations in CO_2 yield between tubing bomb and continuous tests are relatively small. Carbon monoxide yields (0.7-1.3% on daf coal) show similarly small variations which suggests that oxygen elimination as carbon oxides is not significantly dependent on residence time - i.e. tubing bomb tests of 25 and 60 minutes duration give essentially the same yields as the continuous system with its inherent short circuiting.

Data on hydrocarbon gases are less satisfactory, with indicated yields of individual gases varying by up to $\pm$1% vs. daf coal for nominally equivalent feeds and test conditions.

For C_4-C_6 hydrocarbons, consistently higher yields from continuous tests than from tubing bomb tests and the presence of C_6 materal at up to 0.6% of daf coal suggests a systematic sampling error such as foam transport of (nominally) liquid material. However, even for methane, C34 and C35 show significantly different yields (1.9% and 3.4% respectively).

These results present a problem with respect to establishing hydrocarbon yield distribution, since to be conservative it is necessary to assume that the high range of (undesirable) C_1-C_3 hydrocarbon yields, or ~7.5% for Trench B and 8.6% Trench A, are valid. Results of tubing bomb tests at the average continuous system residence time would support significantly lower C_1-C_3 yields of 3.3% for Trench B and 5.7% for Trench A coal.

4.4.1 Hydrogen Availability from Reforming C_1-C_3 Hydrocarbon Byproduct Gas

Reforming of indicated C_1-C_3 hydrocarbons from continuous tests at a 1:2 mole ratio of O_2:H_2O would generate hydrogen equivalent to 2.5 wt% of daf Trench B coal or 2.9% of Trench A coal. As noted

previously, hydrogen consumption in hydrotreating of recycle solvent showed wide variations (0.5-2.5 g H_2/100 g liquid, see Table 12). For Trench B recycle hydrotreating, average hydrogen consumption was 1.8 g/100 g which corresponds roughly to a hydrogen consumption of 3.7 g/100 g daf coal. For Trench A, corresponding hydrotreating and coal hydrogen consumption are 0.75 and 3.0 g/100 g respectively.

These indicated hydrogen consumption values and potential hydrogen production values from C_1-C_3 hydrocarbon are both dependent on inconsistent gas yields. Thus, it is not possible to make a definitive statement regarding adequacy of C_1-C_3 hydrocarbon byproducts for hydrogen production. The data suggest a slight deficit, on the order of 0.6 H_2/100 g daf coal. Byproduct gas yields from hydrotreating recycle solvent (see Table 11) were in the range of 0.04-0.1 g/100 g liquid or almost two orders of magnitude lower than hydrocarbon gas production from coal. Thus, hydrotreater byproduct gas is not a significant potential source of gas for reforming.

4.5 Product Characterization and Potential Utilization

4.5.1 Net Product

Data presented in Sections 4.3 and 4.4.1 indicates that process hydrogen demand is roughly equivalent to hydrogen available from steam reforming of all C_1-C_4 hydrocarbon gas. In commercial practice, separation for sale of C_2-C_4 gas and purchase of equivalent methane might be preferred, but the minor additional revenue involved is not considered significant for preliminary economic analysis (see Section 4.8).

Thus, it is assumed that all hydrotreater product boiling above 195°C represents recycle solvent as in the test program while the C_5-195°C (pentane-cis decalin) fraction is considered as net product.

The liquefaction raw product, and to a lesser extent the hydrotreater product, contain nitrogen components which are potentially water extractable and separately marketable as chemicals at significantly higher prices than light liquid fuels; such materials would also be very deleterious components of feed to fuel processing units.

Product from the third full recycle run (C37-H31) on Trench A coal was selected for detailed analysis and aqueous acid-base stripping to characterize the C_5-195°C net product.

4.5.2 Net Product Properties and Utilization

Table 15 presents chromatographic analyses of H31 naphtha before/after aqueous acid and alkali stripping; Table 16 presents elemental analyses of the raw and stripped naphtha.

With respect to oxygen heteroatom removal, the phenol and cresol components are removed to >85% and >96% respectively according to chromatographic results (Table 15). Actual removals may be higher, but individual peaks below 0.1% of total response cannot be reliably identified with available equipment. The oxygen contents by difference (Table 16) do not account for the phenol and cresol removed, so must be regarded as of doubtful accuracy, although there is likely to be some residual oxygen content in ethers and other non-extractable species.

For nitrogen species, chromatographic data for pyridine indicate >89% removal. Pyridine is the only nitrogen component normally identifiable by GC in light liquefaction products, and the nitrogen content of pyridine present at 0.91% (Table 16) accounts for 77% of the 0.21% of analytically-determined nitrogen in raw naphtha.

Sulphur is quite effectively removed by hydrotreating, as indicated by the 0.08% S content of raw naphtha. No sulphur components have been identified by GC analysis, so efficiency of sulphur removal by aqueous stripping can only be described as $\geq$75% based on analytical results. The stripped naphtha performed well in the copper strip test (ASTM D130), showing no visible stain after 3 h at 50°C.

With respect to utilization, an obvious direct outlet for stripped naphtha is as a motor gasoline blendstock.

Attractive features include:

- high aromatics content (i.e. useful as an octane enhancer for unleaded fuel)
- acceptable distillation range (some blending of light components would be required to increase vapour pressure)
- acceptable sulphur and nitrogen contents.

The naphtha would not require reforming for use as gasoline, so severe restrictions on sulphur and nitrogen (reforming catalyst poisons) would not apply.

An alternative outlet for the naphtha would be manufacture of benzene or benzene and toluene via the Litol process which has been used commercially on coal derived products (18).

The higher boiling fraction of naphtha, roughly +160°C, would be compatible with diesel fuel but its high aromatic content would result in low cetane number and high smoke index.

4.5.3 Distillation and Coking Behaviour of Product Liquid and "Sludge"

Thermogravimetric analyses of the liquid and "sludge" fractions from run C37 are presented in Figures 4 and 5. TGAs of the hydrotreated product (+190°C fraction of H31) and C37 residue are presented in Figures 6 and 7.

The liquid hydrotreater feed (ibp ~70°) is primarily light and middle distillate; gross coke yield or approximate Conradson carbon content 5.3% as shown in Figure 4. Ash content is negligible.

"Sludge" is defined as THF-extracted material which - after THF removal by distillation - is insoluble in the primary liquid product at ambient temperature. The staged heating (see Figure 5) to 325°C, 525°C and 900°C is designed to model the (increasingly severe) operations of distillation, low- and high temperature coking.

The sludge fraction, separated by centrifugation, contains 54.9% of material distillable to 325°C. This is considered to be principally occluded liquid phase. The sludge precipitate is very fine and - from its slow settling behaviour - is apparently only slightly different in density from the liquid. Thus, recovery of liquid by centrifugation is incomplete. The +325°C fraction breaks down to 76.7% coker volatiles and 24% gross coke on heating to 525°C. The higher coke yield from the +325°C fraction of sludge (~50%) compared to liquid (~45%) is consistent with a higher preasphaltene content. It should be noted that the ambient temperature insolubility definition of sludge is quite arbitrary.

It is considered probable that a major part of the +325°C residue from the liquid phase represents asphaltene/preasphaltene material soluble in the aromatic solvent "oil" at elevated temperature.

With or without its distillate content, the sludge phase would not be readily marketable as an oil fuel due to its high insoluble content and consequent incompatibility with virgin residual oils.

The negligible ash content of sludge would make it a premium feedstock for needle coke production, but feedstock prices for this application are generally in the same range as heavy fuel oil.

For the binder pitch market, assuming THF solubility can be equated to quinoline solubility, the pitch (+325°C) content of sludge has a high "beta resin" (benzene insoluble, quinoline soluble) content. This property, combined with high fixed carbon and low sulphur and ash contents would make the material potentially attractive as a component of aluminum reduction electrodes. Removal of contained light oil by distillation or solvent treatment would be required.

Subject to confirmation of predicted properties by testing of a bulk sample, this market could provide a direct outlet for part or all of the solvent insoluble fraction of sludge which represents a significant (10-12%) recycle load on the primary liquefaction operation. Depending on its use-specific properties, binder pitch may be valued at parity with or even above light crude oil (6). This (potential) high market value plus the possibility of reducing the recycle loading suggest that testwork on sludge as a binder pitch should be included in any future assessment of Hat Creek coal liquefaction.

Other markets for heavy aromatic oil such as carbon black manufacture are unattractive due to price (roughly equivalent to heavy fuel oil) or inaccessible due to high insoluble content.

4.6 Chemical Stability of Tetrahydrofuran

Initial tests on chemical stability of THF in contact with liquefaction materials were done in sealed pressure tubes at elevated temperature. Results of 96 h tests at 105°C (Table 17) indicated quite small losses which were considered potentially acceptable for use of THF as a practical separation solvent with (virtually) quantitative recycle.

On this basis a series of larger scale tests were performed in Soxhlet apparatus with THF-benzene-residue water mixture to assess the chemical stability of THF under conditions of repeated atmospheric pressure distillation. In this test approach it was expected (from the 6% THF loss in contact with water over 96 h at 105°C, Table 17) that hydrolysis to 1,4 butane diol would be the main mechanism of loss. Quantitative estimation of loss was done by GC analysis of 1,4 butane diol in distillation residue (water-residue slurry).

Test results showed a THF decomposition rate of 0.15 $\pm$ 0.05% per distillation which was considered unacceptably high for large scale utilization due to the prohibitively high cost of THF.

4.7 Solid Waste Disposal

Typical solid "waste" or residue from THF extraction contains ~80% "ash" (see Figure 7) and has a calorific value of ~6 MJ/kg. Residue sulphur content of 1.2-1.4% in current testwork is principally due to sulphidization of exchanged iron catalyst. Assuming coal beneficiation to ~20 GJ/tonne before liquefaction, process residue will be generated at a rate of ~0.28 tonnes/tonne of coal feed or 2800 tonnes/day for a conversion plant producing 20,000 bbl/day of net naphtha product (see Section 4.8).

Initially, direct landfill disposal was investigated as a low cost option. However, preliminary bioassay test results indicated residue leachate would be toxic to fish (rainbow trout) even at high dilution (100:1, water:solids).

The observed toxicity is tentatively attributed to water soluble heavy phenolics equivalent to ~2 mg/L phenol at the test dilution.

Since the quantity of solid waste is large, of extremely low fuel value and has a significant sulphur content, landfill disposal would be the preferred option for disposal on a cost basis. This would require impoundment in a clay/membrane sealed pond designed to secure essentially zero discharge to groundwater.

The arid climate (30 cm annual average precipitation (7)) and availability of mine overburden clay are favourable factors with respect to stable landfill disposal.

If landfill disposal is not acceptable, a practical but expensive alternative would be combustion in admixture with coal washing rejects (calorific value ~10 GJ/tonne). Mineral matter associated with Hat Creek coal contains substantial quantities of alkaline oxides (3.5% CaO, 1.5% MgO, see Table 2) required for control of sulphur oxide emissions. Alkali content of the residue alone would provide a Ca:S mole ratio of 1.4 which is below the generally accepted level of 2.0 required for 80% retention of SO_x. Thus, blending with coal rejects or purchased limetone would be required. In a 1:1 blend of process residue with coal rejects of calorific value ~10 GJ/tonne, combustion feed would have a Ca:S mole ratio of ~2.2 and a calorific value of 8 GJ/tonne. At a combustion feedrate of 5600/tonne/day (2800 t/d residue + 2800 t/d rejects), gross available power from combustion would be 2000 GJ(t)/h or - at 20% efficiency - 400 GJ(e)/h, or one megawatt (electric) which is ~80% of the estimated plant electric power requirement. In practice, use of such low grade heat

is likely to be confined to space or process heating rather than electrical power generation. Consequently, stabilization of process waste by combustion could represent a significant process cost. Balancing this potential cost is the fact that coal "ash" from combustion at T~800° is a potential source of alumina (7).

Overall, landfill would be the preferred option for residue.

4.8 Economic Analysis

In any economic analysis it is necessary to identify assumptions and bases for comparison. In the case of direct coal liquefaction this presents some difficulties due to the fact that there are no commercially operating plants to provide reference data.

The first and most critical assumption is that the process sequence developed for Hat Creek coal at laboratory scale would be technically viable at a commercial scale. This is considered to be a reasonably safe assumption on the basis of the continuing operation of a 200 t/d pilot plant in the Federal Republic of Germany (8). This plant operates under significantly more severe conditions of temperature and pressure than are required for Hat Creek coal, and process data for this demonstration unit have been satisfactorily correlated with laboratory data of the type developed for Hat Creek coal liquefaction.

Raw material availability and cost are obviously also important; one major result of the oil shocks of the 1970's is a general awareness of the risks of heavy dependence on an imported commodity for which the price is subject to a large measure of political control.

Mineable coal resources at Hat Creek are in excess of 2×10^9 tonnes, based on drilling at 100 m centers; continuation of the deposit beyond the zone of intense exploration (in support of mine planning) indicates a total mineable resource of $\sim 6 \times 10^9$ tonnes.

Considering only the 2×10^9 tonnes of fully proven reserves, conversion data presented in this study indicate that this is equivalent (at 63% conversion on calorific value basis) to ~3.6×10^9 bbl of oil equivalent. It is interesting to note that this quantity of "oil" corresponds to the 60% of remaining "proven" Canadian reserves of crude oil (9). This comparison is not intended to suggest that liquid hydrocarbons from Hat Creek coal will be cost competitive with conventional oil, but rather to establish the magnitude of the resource in relation to conventional petroleum liquids.

In addition to its size, the deposit is advantageously located with respect to:

surface transport - adjacent to the B.C. Rail main line and a provincial highway

pipeline transport - ~16 km from the natural gas and oil lines in the Thompson River valley

electric power - adjacent to main transmission lines

social infrastructure - the deposit is within commuting distance of Cache Creek and Ashcroft (combined population ~5,000) and smaller communities are located close to the site.

Water supply at the site is minimal, but the Thompson River could provide up to 10,800 m^3/day via a 16 km pipeline without interfering with the river's fisheries capability (7).

Convenient access to the port of Vancouver is significant in respect to both construction cost and product marketing. Shop or overseas fabricated process units could be transferred to rail for shipment direct to the site. With respect to product marketing, the B.C. lower mainland is a large, concentrated market area and the port has all necessary facilities for overseas exports. Pipeline connections to Washington state are also available to access the U.S. market.

Economics of a resource development by the private sector are substantially affected by tax factors which are not readily assessable except by industry specialists. For this reason, the main approach to economic assessment of direct liquefaction of Hat Creek coal has been to compare results of a published industrial study (10) with estimated data for a Hat Creek development. Tables 18 and 19 compare major unit operations and economic parameters. Similarities between the two potential developments are considered sufficient to allow at least qualitative extrapolation of the venture analysis to Hat Creek. The published study deals with indirect liquefaction by gasification, methanol synthesis and conversion of methanol to motor gasoline and liquefied petroleum gas (LPG). The study deals with a B.C. location, so it is reasonable to expect that taxation would be equivalent for either development.

4.8.1 Capital Costs

With reference to Table 19, capital investment for Hat Creek has been estimated using the high end cost ($60,000/daily barrel of nominal capacity) of a range of estimates published for Canadian situations by the National Coal Conversion Committee (NCCC) (9). Choice of the high end estimate for a base case is considered reasonable due to the high ash content of Hat Creek coal. The estimate is somewhat lower than the $78,300/daily barrel calculated from the published indirect liquefaction study which reflects the cost of air separation as well as the requirement for three major unit operations (gasification,

methanol synthesis and methanol-to-gasoline conversion) as opposed to two stages (liquefaction and hydrotreating) in direct liquefaction. For general comparison purposes, it is worthwhile to note that the total investment for tar sand based synfuels plant is on the order of $100,000/daily barrel (9).

4.8.2 Manufacturing Cost

4.8.2.1 Coal

The NCCC study included capital cost of an on-site mine in the overall cost estimate. This has not been done in the present analysis, which should make the high end estimate conservative. For the present analysis, mine capital and operating costs are considered to be included in the coal feedstock cost of $15/tonne. This value is justified by a 1984 site specific cost estimate prepared for B.C. Hydro. The most recent mining cost estimate for Hat Creek coal is $(C, 1984)9.08/tonne of product coal. This estimate by B.C. Hydro staff (5) is based on a mining rate of 9.7×10^6 tonne/year, a 4% real interest rate (ie., inflation + 4%) and includes the 3.5% provincial coal royalty. Quality of projected coal product is presented in Table 2.

The $9.08 cost is equivalent to $0.65/GJ (from calorific value, Table 2). Coal washing is not included, as the plan assumes a minesite power plant using run of mine coal.

The estimate of $15/tonne for plant feed at a calorific value of 20 GJ/tonne allows for cleaning costs and ~10% loss of calorific value in cleaning of the coal during addition of ferrous sulphate catalyst. No correction has been made for the reduced scale of mining (~4×10^6 tonnes/year vs. Hydro cost based on 9.7×10^6 tonne/year).

For an external comparison, open pit thermal coal in the East Kootenay area of B.C. is currently mined at costs $\leq$\$20/tonne for washed coal fob minesite (11). A lower cost is realistic for Hat Creek in view of seam thickness (up to 300 m with minor partings), low stripping ratio and large scale of operations.

The cost (\$15/tonne or \$0.75/GJ) for Hat Creek coal is significantly higher than the cost of coal rejects to the indirect liquefaction plant (\$6/tonne or \$0.27/GJ).

4.8.2.2 Electric Power

The power cost estimate for the indirect liquefaction study relates mainly to the air separation plant. This operating cost estimate has been reduced to \$10 x 10^6/year for direct liquefaction. Power costs associated with coal mining are included in the mining cost estimate.

4.8.2.3 Catalyst and Royalty

The indirect liquefaction estimate for catalysts and royalties is high (\$35 x 10^6/year) reflecting the 2 catalytic operations and royalties on the proprietary zeolite catalyzed methanol conversion to gasoline.

For direct liquefaction, the iron exchange using ferrous sulphate could be a significant cost. Bulk ferrous sulphate is readily available fob ocean ports at \$30/ton (12) and could be transported by rail to the site at a convervative cif cost of \$(C)60/tonne. For addition of 1% iron to coal (dry basis), the material cost would be \$3/tonne of cleaned coal or \$10 x 10^6/year. A more likely scenario would be purchase of waste pyrrhotite (FeS) from the Sullivan mine near Kimberley and local manufacture of ferrous sulphate. This would reduce the cost to $\leq$\$1/tonne of coal or $\leq$\$3.3 x 10^6/year.

The cost of hydrotreating catalyst is more difficult to define since the useful life of a Ni-Mo-Al_2O_3 catalyst in coal liquid treatment is not known. Analogous oil industry experience in treatment of solids-contaminated, asphaltic material indicates that a one year lifetime with capability for at least one regeneration is a reasonable estimate. An initial charge of 500 tonnes of catalyst would cost ~\$9 x 10^6. Assuming that the scrap value of the catalyst would balance the cost of a single regeneration, this indicates an annual hydrotreating catalyst cost of ~\$5 x 10^6. Thus, overall annual catalyst costs would be in the range of \$8-15 x 10^6/year with the lower cost being more probable.

In an initial commercial development of direct liquefaction at Hat Creek, it seems likely that a company or consortium holding process technology and related patents would be involved in equipment specification, design and manufacture. On this basis, some indeterminate process royalty might have to be negotiated. This is not to suggest that any proprietary technology has been used in current testwork; the concept of using donor solvents produced by hydrotreating product is certainly well known. However, acquisition of the engineering/process expertise to apply this concept at a commercial scale would probably require participation of a firm holding patents or licenses and requiring a royalty. On this basis, it is considered reasonable to make allowance for process royalty. In the absence of specific data, the allowance made for the indirect study covering catalysts and royalties seems a reasonable first approximation.

Thus, Table 19 includes an annual charge of \$35x10^6 comprising \$8-15x10^6 for (somewhat) predictable catalyst cost with the balance for any applicable royalties.

4.8.2.4 Labour

Considering manufacturing labour, comparison of the major unit operations of the two processes (Table 18) indicates a similar degree of overall process complexity. On this basis, the operating/maintenance staff level of 700 used in the indirect liquefaction study is considered appropriate. The division of (roughly) 300 operating/400 maintenance staff is also considered appropriate in view of the severe conditions (pressure-temperature-corrosion-erosion) required for both processes. Labour rates for southeast B.C. can be reliably used for Hat Creek, since both areas are well serviced and accessible; ie., no remote area premiums would be required to attract a stable, qualified staff to either area.

4.8.3 Revenue

Revenue for the direct liquefaction is based on $40/bbl (vs. ~$35 current crude price) on the basis of its 100% light distillate content and recoverable phenolics (~3 wt %, marketable at 1.5-2x fuel prices. At $40/bbl for naphtha, the ratio of refinery gasoline sale price to feedstock cost would be ($47.60/40)=1.19 compared to 1.37 for use of crude oil. The lower ratio is considered to be jusfified by higher volumetric yield of gasoline and complete absence of heavy products which are only saleable at or below crude oil prices.

If the direct liquefaction product is assumed to be eligible for the same (conceptual) tax remission subsidy as is assumed for the indirect liquefaction base case (10), then addition of a final refining stage to produce motor gasoline would be advantageous. Assuming a 95% volume conversion to gasoline, and the (indirect case) projected gasoline price (refinery net) of $48.24/bbl or $0.3054/L, total revenue for the direct liquefaction case becomes $916,560/day or $302 x 10^6/year. This adjustment - as indicated in Table 19 - makes total revenues and revenues vs. capital or operating costs significantly more attractive than for the indirect liquefaction case.

4.8.3.1 Rate of Return and Payback

The estimated manufacturing costs for both processes are quite similar; differences are in capital costs ($1.42 x 10^9 for indirect vs. $1.2 x 10^9 for direct liquefaction) and projected revenues.

For capital cost, the difference favours direct liquefaction by ~15%.

For revenue, the direct liquefaction shows a range from slightly below indirect for the no subsidy case to significantly higher in the case of a gasoline tax remission subsidy.

For indirect liquefaction, the base case with subsidy shows a 17.5% discount cash flow (DCF) rate of return and 5 year (post startup) payback. In the words of the author of the study, "At 17.5% the base case DCF rate of return would be good enough for the project to be seriously considered...." (10).

For the direct liquefaction, (no subsidy) case, revenue is slightly lower both in absolute value and as a percentage of operating cost.

However, if net revenue is compared to capital cost (ie., simple return on investment) the cases become virtually identical:

$$\text{Percent Return on Investment} = \frac{\text{profit}}{\text{equity*}} \times 100$$

Indirect Liquefaction, 7.4%
Direct Liquefaction, 7.4%

*equity in this case being capital cost

Thus, while calculation of rate of return and payback for the direct liquefaction case would require access to the model used for the direct case, the similarities between the two cases suggest that the values would be similar to well within the limits of accuracy of the estimates involved. Thus, the direct liquefaction case can be considered by analogy as "...good enough for the project to be seriously considered."

If the subsidy assumed in the indirect base case is applied to direct liquefaction, the return on investment for the direct liquefaction case increases to 10.4%.

Thus, it would appear that from a point of view of government return on its investment (subsidy), the direct liquefaction case would be preferred. In both cases, the subsidy goes into net revenue which - after payback - becomes taxable. However, the higher return on investment for the direct liquefaction case suggests a faster payback and higher profitability.

4.8.4 General Evaluation

The indirect liquefaction of East Kootenay coal rejects represents a special case in two respects:

- the availability within a confined geographic area of a large quantity of coal rejects which - due to transport economics and local power supply costs - is presently unmarketable and could thus be made available for conversion at the very low cost of $6/tonne or ~$0.66/GJ.

- availability of large volumes of low cost grid power at a rate ($0.02/kwh) which could not be matched by new power development. It should be noted that the differential electric power input between the direct and indirect

liquefaction cases is equivalent to combustion of 3030 tonnes of coal/day at a 25% coal to busbar efficiency, thus increasing coal reject use by 31%. (The use of a low (25%) efficiency is considered reasonable for the East Kootenay coal which is ~40% ash and low in volatile matter so poor burnout can be expected). Burning of coal rejects to generate this power might be feasible at a fuel cost of $0.004/kwh(e), but a thermal coal product from other area mines at $20/tonne and 28 GJ/tonne would offer no practical possibility of matching the $0.02/kwh(e) power cost used in the study.

The Hat Creek direct liquefaction concept is also to some extent a unique opportunity due to the large single site reserves, very thick seams and low stripping ratio. These factors plus the fact that (relatively) valuable agricultural land would not be involved allow the slightly lower coal supply cost of $7.50/bbl of product compared to the $9.75/bbl feedstock cost reported for open pit plains coal (9).

In comparing the two conceptual projects on an equivalent (ie., equal subsidy) basis, it appears from the present analysis that the Hat Creek direct liquefaction approach has a significantly better return on investment and thus would be the preferred candidate for subsidy on a financial basis. Either project would be highly sensitive to a significant reduction in product selling price, but the higher indicated ROI for Hat Creek would provide some margin of protection. For example, at a constant subsidy (tax remission) of $0.115/L of gasoline product, net revenue for the Hat Creek project could be maintained at the base case level with a refinery net motor gasoline price of $0.265/L. Using the price relationship (1.37 x crude acquisition cost) indicated in the indirect liquefaction study between crude oil and motor gasoline, this corresponds to a crude oil price of $0.193/L or $(C)30.73/bbl or $(US)23.05/bbl; $(C)1 = $(US)0.75.

Obviously, the future price of crude oil in a specific currency is a very difficult item to predict. However, the recent (13) range of world oil prices is at least a point of reference. All prices (OPEC, non OPEC, world and US imports) fall in the fairly narrow range of $(US)26.99-27.96/bbl. All have also declined in the preceding 12 months by an unweighted average of 4.2%.

Thus, for the Hat Creek project with subsidy, the oil price of $(US)23.05/bbl to meet the 17.5% DCF rate of return and 5 year payback would allow a 16% reduction in world oil price in U.S. funds at constant exchange rates vs. Canadian funds.

Such a decrease is possible, but such a low crude price would certainly make some current production uneconomic in Canada and elsewhere while leaving the Hat Creek project viable.

On this basis, it would appear that direct liquefaction of Hat Creek coal is potentially viable at current liquid fuel prices without subsidy, and is a more attractive proposition for government subsidy than indirect liquefaction even at a site favoured by very low coal and electric power costs.

Finally, it is worthwhile to consider projected costs for exploration/development/production from conventional (offshore) and heavy petroleum resources.

Heavy Alberta-Saskatchewan crude requires investment of $(C)76,000-104,000 per daily barrel, while surface mined tar sand - as indicated in the indirect liquefaction study - has required a total investment of $(C, 1983 basis)100,000. Projections for Grand Banks and Beaufort Sea offshore light crude range from $81,000 to $112,000/daily barrel. From these data, coal liquefaction appears to be a fully competitive technology (9).

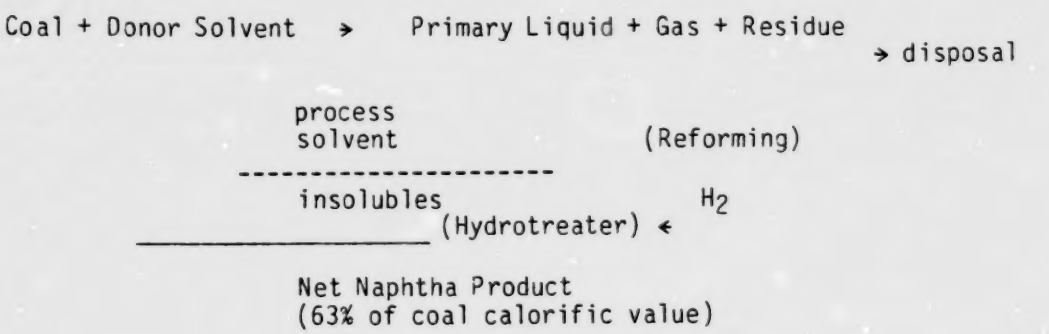
It is noted that a non published and non-accessible (14) industrial critique of data presented in reference 10 has concluded that inadequate cost and contingency allowances invalidate the published data.

To whatever extent the critique of published data is valid, inferred costs for Hat Creek coal direct liquefaction may also require improved revision. However, until the industrial review is available, published data of references 9 and 10 are considered acceptable bases for estimation.

5.0 CONCLUSIONS

5.1 Technical Results

Results of the current study confirm that continuous direct liquefaction of Hat Creek coal is feasible in the operational sequence:



Near optimal conditions for liquefaction include:

solvent T:N $\geq$3
residence time ≈30 minutes (multistage to avoid short circuiting)
temperature = 440°C
pressure ≈14 MPa

Use of a ferrous sulphate coal pretreatment is advantageous with respect to both conversion and reduction of raw coal ash content.

The primary liquid-residue separation method (THF extraction) used in this study does not appear to be practical for commercial application due to THF loss. Further work (see below, Recommendation) would be required to demonstrate comparable separation efficiency by developmental processes.

For primary liquid hydrotreating, acceptable (non optimized) conditions are analogous to conventional petroleum industry hydrotreating operations with respect to catalyst (sulphided nickel-molybdenum), temperature (310-375°C) and hydrogen overpressure. The high hydrogen demand precludes use of downflow fixed bed hydrotreating, while the upflow fixed bed method used in this study has not been commercially applied. The test-indicated beneficial effects of high hydrogen flowrates suggest that ebulliated bed hydrotreating analogous to existing commercial processes for heavy oil (15) will prove to be the method of choice for primary liquid upgrading.

Current results indicate that net product will be mainly or exclusively naphtha suitable after stripping for direct use as motor gasoline blendstock.

Net product might also include a light distillate fuel oil depending on recycle solvent volume requirements.

Phenol and cresol, separated by caustic extraction, are potentially significant components of the net product, and could be marketed directly as petrochemicals.

5.2 Economic Assessment

Results of comparative economic analysis indicate that direct liquefaction of Hat Creek coal is less costly than indirect liquefaction of East Kootenay reject coal, even though the latter material has a significantly lower feedstock cost.

To the limited extent that results of the economic analysis can be considered quantitative, it appears that direct liquefaction of Hat Creek coal could be commercially attractive (i.e. $\geq$17.5% DCF) at a crude oil price of about $(C)31/bbl if a subsidy in the form of gasoline tax remission is assumed. Similarly, favourable comparative economics vs. offshore and frontier conventional oil prospects are indicated.

In the short term, downward trends in international fuel prices are a major deterrent to commercial or even R & D investment in coal conversion. For the indeterminate future, however, results are considered to indicate that direct liquefaction of Hat Creek will become viable when liquid fuel prices increase marginally over current (constant dollar) levels and the predictable long term uptrend in fuel prices is re-established due to depletion of low cost reserves.

5.3 Environmental Assessment

In terms of social environment, installation of a mine and liquefaction plant at Hat Creek would increase regional population significantly, thus requiring adjustments in social infrastructure. Direct employment in a mine (~150) and liquefaction plant (~700) would give rise to a similar number of secondary and service jobs in the local economy. Previous studies indicate that health and education services would require minimal expansion to accommodate this growth.

Land and water use would be similar in pattern to documented requirements for previous development concepts (7), although at a smaller scale initially due to the smaller scale of direct liquefaction operations compared to planned thermal power and/or indirect liquefaction development concepts.

Atmospheric emissions from operations, particularly calcination of solid waste, would have to be controlled with particular respect to SO_x emissions due to increased sulphur content of waste or raw coal from iron sulphide catalyst.

Zero discharge of aqueous waste is expected to be a practical option due to the low regional precipitation and substantial water immobilization by mine waste. Impermeable lining of process residue (waste) impoundments would be essential for the preferred landfill disposal option.

6.0 RECOMMENDATIONS

6.1 Liquid-Residue Separation

The liquid-residue separation component of the process scheme is the only one which has not been demonstrated at bench scale under practical conditions analogous to petroleum processing operations. Testing of advanced separation technologies such as the Sandwell-Centrex process and the various critical solvent technologies should be undertaken to determine whether "liquid" product recoveries equivalent to THF soluble material can be obtained.

6.2 Coal Beneficiation

Current data on ferrous sulphate treatment and conventional washing of Hat Creek coal indicate a lower limit of ~24% ash in liquefaction process fuel. This high ash content causes problems in both liquefaction and liquid-residue separation.

Selective oil agglomeration processes for low rank coal are being developed in Canada (16,17), although little quantitative information has been published. Application of these processes to Hat Creek coal should be investigated, since the potential for improvements in liquefaction (lower reactor volume, reduced slurry viscosity) and liquid-residue separation is quite significant. Also, developmental agglomeration processes are understood to involve use of high sulphur heavy oil or bitumen fractions which would reduce or eliminate consumption of sulphur for iron catalyst activation.

6.3 Co-processing

Use of heavy oil or bitumen for selective agglomeration of Hat Creek coal would necessarily involve co-processing of the oil-coal agglomerate product with the above-noted advantage of iron catalyst activation by oil phase reactive sulphur. Bitumen and heavy oil fractions are known to be responsive to donor solvent upgrading, so primary liquid production per unit of reactor volume should be improved.

On this basis, subject to demonstration of useful coal beneficiation by agglomeration technology, co-processing testwork is recommended to investigate several parameters including iron catalyst sulphidization, liquid and gas yields and liquid-residue separation.

6.4 Binder Pitch Manufacture

The THF soluble "sludge" insoluble in process solvent at ambient temperature is a significant (9-12%) recycle component in the tested liquefaction sequence. Electrode grade binder pitch is a potential direct market outlet for this material at or above crude oil price. Any future study should include material evaluation for this market due to its potential for reducing processing costs.

6.5 Pilot and Demonstration Plant Testing

In view of the potential for process improvements (selective agglomeration, co-processing, direct marketing of "sludge"), the need for further bench scale liquid residue separation studies, and current trends in fuel prices, larger scale process testing is not considered to be cost effective at present.

Correlation between bench and pilot scale test data for other coals using analogous donor solvent processes indicate that scaleup can be achieved. Thus, any available research or development funds would be more effectively devoted to investigation of process improvements at the batch or continuous bench unit scale.

R.O. McElroy
Manager, Fuel Programs
Chemical Technology Division

D.W. Duncan
Head
Chemical Technology Division

7.0 REFERENCES

1. B.C. Research, Continuous Liquefaction of Hat Creek Coal, Project Report 2-02-657, DSS File 15SQ.23440-3-9060, December, 1984.

2. B.C. Research, Project Proposal - Liquefaction of Hat Creek Coal, DSS 15SQ.23440-3-Energy Conversion-X, November, 1983.

3. Hatswell, M.R., Jackson, W.R., Larkins, F.P., Marshall, M., Rash, D. and Rogers, D.E., Hydrogenation of Brown Coal.2. Iron Based Catalysis, Fuel 62, March, 1983, p33C.

4. Gutierrez, L., Jezko, J., and McElroy, R.O. Direct Liquefaction Behaviour of B.C. Coals, Coal-Phoenix of the '80's, Proceedings 64th CIC Coal Symposium, Halifax (1981).

5. McCullough, T., B.C. Hydro, Personal Communication, August, 1985.

6. Stokes, C.A. and V.J. Guereio, Feedstocks for Carbon Black, Weedle Coke and Electrode Pitch, Erdol und Erdgas, 1, 38, January, 1985, p.31 ff.

7. B.C. Research and Dolmage Campbell and Associates Ltd., Preliminary Environmental Study of the Proposed Hat Creek Development, B.C. Hydro and Power Authority, 1975.

8. Anon., The Coal Hydrogenation Plant at Bottrop, in Handbook of Synfuels Technology, R.A. Meyers ed., McGraw Hill, 1984, p.1-101 ff.

9. National Coal Conversion Committee, Position Paper on Coal Liquefaction in Canada, Energy, Mines and Resources Canada, April, 1985. (DSS Contract 03GR.23218-3-0014).

10. Taherzade, M., Coal to Gasoline - Is It Economic? Paper presented at CANMET Coal Conversion Contractors Meeting, Calgary, Nov. 14-16, 1984.

11. Evans, J.B., Esso Resources Canada Ltd., Personal Commnication, November, 1984.

12. Chemical Marketing Reporter, December 2, 1985.

13. Oil and Gas Journal, June 17, 1985.

14. Johnson, W.M., B.C. Ministry of Energy, Mines and Petroleum Resources, Personal Communication, October, 1985.

15. Li, A. and D. Lin, "Scaleup Performance and Thermal Stability Analysis of H-Oil and H-Coal Ebullated-Bed Reactors", Second World Congress of Chemical Engineering, Montreal, Canada, October, 1981.

16. Ignasiak, L., Alberta Research Council, Personal Communication, December, 1985.

17. Mikhlin, J., SNC Ltd., Personal Communication, Sept. 1985.

18. Dufallo, J.M., W.A. Schwartz and D.C. Spence, The Modified Litol Process for Benzene Production, in Handbook of Synfuels Technology, R.A. Meyers, ed. McGraw Hill, 1984.

TABLE 1

PROXIMATE ANALYSES AND CALORIFIC VALUES OF TEST COALS

COAL (code)	PROXIMATE ANALYSIS (%)							CALORIFIC VALUE (As Tested)	
	As Tested				Dry Basis				
	Moisture	Volatile Matter	Fixed Carbon	Ash	Volatile Matter	Fixed Carbon	Ash	Btu/lb	MJ/kg
Trench A (C-03-2)	21.8	25.5	15.8	36.5	32.8	20.3	46.9	3908	9.1
Trench A FeXC* (C-03-2)	8.5	33.6	29.5	28.2	36.8	32.3	30.9	6580	15.3
Trench B (C-04-2)	20.5	27.6	23.3	28.0	34.7	30.5	35.2	5700	13.3
Trench B FeXC (C-04-2)	3.9	38.5	30.5	27.2	40.2	31.9	28.4	7050	16.4

*Ion exchanged with ferrous sulphate solution

TABLE 2

PROJECTED RUN OF MINE HAT CREEK COAL QUALITY

		AS PRODUCED	DRY BASIS
Moisture	(%)	23.5	0
Volatile Matter	(%)	25.2	32.9
Fixed Carbon	(%)	25.7	33.6
Ash	(%)	25.6	33.5
Gross Calorific Value	(MJ/kg)	13.85	18.06
Organic Content Analysis (%)	C	-	46.1
	H	-	3.7
	O	-	15.3
	N	-	0.92
	S	-	0.51
	Cl	-	0.03
Hardgrove Grindability Index		45	
Ash Analysis (%)	SiO_2	-	52.6
	Al_2O_3	-	28.1
	TiO_2	-	1.0
	Fe_2O_3	-	8.5
	CaO	-	3.5
	MgO	-	1.5
	Na_2O	-	2.2
	K_2O	-	0.62
	P_2O_5	-	0.17
	SO_3	-	1.75
	Mn_3O_4	-	0.16
	V_2O_5	-	0.06
Sulphur Forms (%)	Pyrite		0.01
	Organic		0.28
	SO_4		0.01

TABLE 3

IRON CONTENTS OF TEST COALS BEFORE AND AFTER ION EXCHANGE

COAL (code)	Fe (wt. %)		Fe UPTAKE (wt. %)
	As Tested	Dry	
Hat Creek Trench A (C-03-2)	1.25	1.61	-
Hat Creek Trench B (C-04-2)	0.82	1.02	-
Hat Creek Trench A (Fe-ex C-03-2)	2.44	2.65	1.04
Hat Creek Trench B (Fe-ex C-04-2)	2.04	2.12	1.10

TABLE 4

HYDROTREATER TEST DATA ON RAW CREOSOTE

RUN	FEED RATE Liquid kg/hr	FEED RATE Hydrogen sl/min	STEADY STATE CONDITIONS Pressure MPa	STEADY STATE CONDITIONS Temperature °C	TIME h	GROSS LIQUID PRODUCTS kg	GROSS LIQUID PRODUCTS % of feed	DISTILLATION wt. % ibp-180°	DISTILLATION wt. % 180-200°	DISTILLATION wt. % +200°C	T/N RATIO Initial	T/N RATIO Product (+200°C)
H17	1.12	2.5	10.2	310	4.00	6.05		18	16	66	0.015	0.92
H18	1.21	2.5	10.2	325	5.00	6.10	101	20*	11**	69	0.015	0.62
H19	1.16	5.0	13.8	325	6.52	7.31	97	15	17	68	0.015	3.40
H20	1.14	2.5	13.8	325	6.00	7.07	104	16	14	70	0.015	0.85
H21	1.80	5.0	13.8	325	6.17	10.85	98	24*	7**	69	0.015	0.54
H22	1.73	5.0	13.8	325	5.50	9.97	105	20	7	73	0.015	1.32
H23	1.77	5.0	13.8	325	5.87	10.02	97	24	9	67	0.015	1.03
H24	1.77	5.0	10.2	325	5.00	8.83	100++	31 18+	2 7+	67 75+	0.015	1.63
H32	1.77	5.06	13.8	325	5.58	6.15	100.6++	20	16	64	0.015	4.08

* ibp-185°C
**185°-200°C
+ redistilled with reflux head
++reactor drained after reaction

TABLE 5

TRENDS IN TOTAL CONVERSION WITH FeXC CATALYST AND INCREASING SOLVENT DONOR CONTENT*

COAL	SAMPLE CODE	ADDITIVE	SOLVENT T:N	TOTAL CONVERSION (wt% daf)
Trench A	C-04-2	Nil	1.0	81.3
	C-04-2	Nil	4.5	82.0
	C-04-2	CS_2	4.5	81.8
	C-03-2	FeXC, CS_2	4.5	88.7, 88.1
	C-03-2	FeXC, CS_2	7.5	90.6
Trench B	C-04-2	Nil	1.0	77.2, 79.5
	C-04-2	Nil	4.5	85.1
	C-04-2	FeXC, CS_2	4.5	89.8

Tubing Bomb System, T = 440°C, t = 25 min, initial P_{H2} = 7 MPa, solvent:coal = 3

TABLE 6

HYDROTREATER TEST DATA ON RETREATMENT OF
HYDROTREATED CREOSOTE, +200°C FRACTION

RUN	FEED RATE Liquid kg/h	FEED RATE Hydrogen sl/min	STEADY STATE CONDITIONS Pressure MPa	STEADY STATE CONDITIONS Temperature °C	TIME AT STEADY STATE h	GROSS LIQUID kg	GROSS LIQUID % of feed	DISTILLATION wt. %		T/N RATIO Initial	T/N RATIO Product (+190°)
H25	2.20	5.0	13.8	325	4.47	9.43	*	-195° / 16	+195° / 84	1.02	4.50
H26	2.17	5.0	13.8	325	5.40	12.16	100**	-190° / 20	+190° / 80	1.02	1.70

* reactor not drained
** combined H25 + H26, reactor drained after reaction

TABLE 7

COMPARISON OF PRODUCTS FROM FIRST AND SECOND STAGE CREOSOTE HYDROTREATING

Run	H24	H25
Feed	Creosote	Hydrotreated Creosote
Product Distillation		
<195°C (wt. %)	16	25 (<200°C)
>195°C (wt. %)	84	75 (>200°C)
Product Component Analyses (peak area %)		
Toluene	0.97	0.59
m,p-xylene	2.86	1.76
o-xylene	1.53	0.94
phenol	3.09	2.19
indan	17.00	12.35
cis decalin	0.54	0.69
p,m-cresol	0.36	0.29
trans decalin	0.08	0.24
tetralin	13.63	22.36
naphthalene	8.38	4.38
9,10-dihydrophenanthrene	0.47	2.44
phenanthrene	2.11	2.20
anthracene	0.17	0.37
pyrene	0.74	1.08
total BTX	5.36	3.29
total decalins	0.62	0.93
T+N	22.01	26.74
T:N Ratio	1.63	5.1

TABLE 8

HYDROTREATER TEST DATA ON CONTINUOUS LIQUEFACTION PRODUCTS

RUN	LIQUID	FEED RATE Liquid kg/hr	FEED RATE H_2 sl/min	STEADY STATE CONDITIONS P (MPa)	STEADY STATE CONDITIONS T (°C)	TIME AT STEADY STATE h	LIQUID PRODUCT kg	LIQUID RECOVERY	DISTILLATION wt. %		T/N RATIO Initial	T/N RATIO Product
H27	C34+C30 Trench B	1.98	5.02	13.8	350	4.53	8.64	100	-180° 14	+180° 86	ND*	11.55
H28	C33+C32 Trench A	1.97	5.02	13.8	350	3.27	6.79	100	-180 8	+180 92	0.67	10.98
H29	C35 Trench B	1.99	5.02	13.8	350	3.65	7.28	100	-180 9	+180 91	1.74	18.35
H30	C36 Trench A	2.09	5.03	13.8	350	2.70	5.78	102	-190 10	+190 90	1.53	15.39
H31	C37 Trench A	1.99	5.06	13.8	350	2.08	4.19	99.3	-180 9	+180 91	2.06	19.76

*Not Determined.

TABLE 9

GAS PRODUCTION
HYDROGENATION OF COAL LIQUEFACTION PRODUCTS

	Gas Production (g/100 g)				
Component	H27	H28	H29	H30	H31
CH_4	0.013	0.023	0.016	0.019	0.011
C_2H_6	0.025	0.032	0.029	0.030	0.012
C_3H_8	0.049	0.045	0.068	0.067	0.021
i C_4H_{10}	0.012	0.012	0.014	0.016	0.004
C_4H_{10}	0.037	0.033	0.053	0.057	0.019
i C_5H_{12}	0.009	0.006	0.014	0.013	0.004
C_5H_{12}	0.012	0.009	0.020	0.018	0.006
i C_6H_{14}	0.007	0.010	0.007	0.013	0.002
C_6H_{14}	0.006	0.005	0.011	0.023	0.004
$>C_6$	0.032	0.057	0.037	0.086	ND
H_2S	0.029	0.053	0.037	0.017	0.008
CO_2	-	-	-	0.005	0.005
Total	0.023	0.028	0.030	0.036	0.009
C_1-C_3 Subtotal	0.087	0.010	0.011	0.012	0.004

TABLE 10

ULTIMATE ANALYSIS, ATOMIC H/C RATIOS AND COMPOSITION CHANGES
OF SELECTED HYDROTREATED FEEDS AND PRODUCTS

	ULTIMATE ANALYSIS (%)						CHANGES (% of liquid)				NOTES
	C	H	N	O	S	H/C*	C	H	N	S	
RAW CREOSOTE**	88.5	8.1	0.86	1.8	1.0	1.10					
H19 product	88.4	9.5	ND***	ND	0.4	1.29	-0.1	+1.4	0.1	-0.6	
H20 product	89.0	9.9	ND	ND	0.6	1.34	+0.5	+1.8	0.1	-0.4	
H22 product	88.9	10.0	ND	ND	0.5	1.35	+0.4	+1.9	0.1	-0.5	
H23 product	89.0	9.6	ND	ND	0.6	1.29	+0.5	+1.5	0.1	-0.4	
H24 product	89.2	9.9	ND	ND	0.5	1.33	+0.7	+1.8	0.1	-0.5	
H25 feed	89.4	9.6	0.41	ND	ND	1.29					
H25 product	89.0	9.9	<0.2	ND	ND	1.34	-0.4	+0.3	-0.2	ND	
H27 feed	88.9	9.0	0.31	ND	ND	1.22					C30 + C34 Product
H27 product	86.5	9.3	<0.2	ND	ND	1.29	-2.4	+0.3	ND	ND	
H28 feed	89.4	8.8	0.6	ND	ND	1.18					C32 + C33 Product
H28 product	89.1	10.2	<0.2	ND	ND	1.37	-0.3	+1.4	ND	ND	
H29 feed	89.6	9.1	<0.6	ND	ND	1.22*					C35 Product
H29 product	89.4	10.1	0.24	ND	ND	1.36 C34	+0.4	+1.0	ND	ND	
H30 feed	89.8	9.0	<0.2	ND	ND	1.20**					C36 Product
H30 product	88.4	9.9	<0.2	ND	ND	1.34 C34	-1.4	+0.9	ND	ND	
H31 feed	89.9	9.1	<0.2	ND	ND	1.22 C32					C37 Product
H31 product	89.3	9.5	<0.2	ND	ND	1.28	-0.6	+0.4	ND	ND	
H32 product	89.4	10.0	<0.2	ND	ND	1.34	+0.9	+1.9	ND	ND	

* H/C=Hydrogen to carbon atomic ratio
** Feed for H19-H24 and H32
***ND=Not Determined

TABLE 11

HYDROTREATER HYDROGEN CONSUMPTION: CALCULATED FROM LIQUID AND GAS COMPOSITION AND "MEASURED" FROM GAS FLOWS

	H%			gas, g/100 g			H_2O*	CALCULATED** H_2 CONSUMPTION	MEASURED*** H_2 CONSUMPTION
	Feed	Liquid Product	Δ	C_1+C_2	C_3+C_4	H_2S	(g/100 g)	(g/100 g)	(g/100 g)
H19	8.1	9.5	1.4	0.043	0.023	0.62		1.55	0.7
H20	8.1	9.9	1.8	----------ND----------				>1.8	0.4
H21	8.1	10.0	1.9	0.032	0.017	ND†		>2.0	0.1
H22	8.1	9.6	1.5	0.044	0.024	ND		1.7	0.3
H23	8.1	9.9	1.8	0.097	0.045	ND		2.0	0.3
H24	8.1	9.9	1.8	0.043	0.024	0.62		2.0	0.6
H25	9.6	9.9	0.3	0.064	0.068	0.59		0.4	0.2
H27 (C33 product TrA)	9.0	9.3	0.3	0.038	0.098	0.029		0.4	0.2
H28 (C34 product TrB FeXC)	8.8	10.2	2.4	0.055	0.089	0.053		2.5	0.1
H29 (C35 product TrB FeXC)	9.1	10.1	1.0	0.035	0.13	0.037		1.1	0.1
H30 (C36 product TrA FeXC)	9.0	9.9	0.9	0.048	0.14	0.017		1.0	0.02
H31 (C37 product TrA FeXC)	9.1	9.5	0.4	0.022	0.045	0.008	1.2	0.5	N/D
H32	8.1	10.0	1.9	0.031	0.045	0.06		2.0	0.7

* Includes emulsified or otherwise non-suspended water from L/R separation
** From liquid and C_1-C_4 gas compositions
***From gas flows and gas phase analyses
† Not determined

TABLE 12

SUMMARY OF CONTINUOUS LIQUEFACTION TEST RUNS* ON HAT CREEK COALS

RUN	COAL	SOLVENT	SLURRY DENSITY	FEED RATE	DURATION	T/N		CONVERSION		MATERIAL BALANCE**	SEPARATION (wt % of product slurry)		
						Initial	Final	Total	Gas		Liquid	Sludge	Solids
			wt %	kg/h	h			wt % daf	wt % daf	%			
C33	Trench A C-04-2	H17-H24 +200°C	26.3	1.44	5.5	0.81	0.55	76.9	N/D	N/D	65.9	5.5	28.6
C34	Trench B FeXC C-04-2	H25 +200°C	39.4	1.37	5.5	2.69	1.29	90.1	19.1	98	71.2	9.9	19.4
C35	Trench B Fe-ex C-04-2	H27 +180°C	37.7	1.15	9.0	6.33	1.87	91.4	20.1	98	76.5	12.4	11.1
C36	Trench A FeXC C-03-2	H29 +180°C	29.4	1.24	6.5	4.77	1.50	89.2	26.6	97	71.1	11.3	17.6
C37	Trench A FeXC C-03-2	H30 +190°C		1.25	5.6	6.78	2.02	89.7	21.9	100	70.6	9.8	19.6

* Conditions: T=440°C, P = 13.8 MPa, H_2 flowrate = 5.0 L/min

**Material Balance = $\frac{\text{wt (product slurry) + (wt product gas)}}{\text{wt (feed slurry)}} \times 100$

TABLE 13

CALORIFIC AND GRAVIMETRIC
CONVERSIONS FROM CONTINUOUS LIQUEFACTION

RUN NO.	CONVERSIONS (%)	
	Calorific Value*	Weight (daf)
C33	80.8	76.9
C34	92.6	90.1
C35	94.4	91.4
C36	90.1	89.2
C37	91.4	89.7

*vs. feed coal

TABLE 14

COMPARISON OF GAS PRODUCTION IN TUBING BOMB
AND CONTINUOUS LIQUEFACTION EXPERIMENTS
Gas Yields (wt% vs daf coal)

	C33		C34		C35	C36			C37	
SLURRY SYSTEM	Trench A		Tr B, FeXC		Tr B, FeXC	Tr A, FeXC			Tr A, FeXC	
	TB/25	CONT	TB/25	CONT	CONT	TB/25	TB/60	CONT	TB/25	CONT
CH_4	2.9	ND	1.8	1.9	3.4	2.3	3.8	2.9	2.7	3.1
C_2H_4	trace	ND	-	trace	trace	-	-	0.1	-	0.1
C_2H_6	1.7	ND	0.9	1.5	2.3	1.4	2.4	2.5	1.7	2.1
C_3H_8	1.5	ND	0.6	1.9	1.8	0.9	1.7	3.1	1.2	1.9
iC_4H_{10}	0.2	ND	0.1	0.3	0.2	0.1	0.2	0.4	0.1	0.2
C_4H_{10}	0.4	ND	0.1	1.1	0.6	0.2	0.5	1.7	0.3	0.7
iC_5H_{12}	0.2	ND	0.1	0.4	0.1	trace	0.1	0.5	0.1	0.1
C_5H_{12}	0.2	ND	0.1	0.6	0.1	trace	0.1	0.5	0.1	0.1
iC_6H_{14}	-	ND	0.1	0.2	trace	trace	trace	0.2	-	0.2
C_6H_{14}	0.1	ND	-	0.3	trace	trace	trace	0.4	-	-
H_2S	trace	ND	trace	0.5	trace	-	-	trace	trace	trace
CO	1.0	ND	0.8	0.7	0.8	1.1	1.1	1.1	1.3	1.2
CO_2	8.1	ND	8.7	8.3	9.8	11.2	12.9	11.7	12.9	12.9
TOTAL	16.3	ND	13.3	17.7	19.1	17.2	22.8	25.1	20.4	21.9
C_1-C_3	6.1	ND	3.3	5.3	7.5	4.6	7.9	8.6	5.7	7.2
C_4-C_6	1.0	ND	0.5	2.6	1.0	0.3	0.9	3.3	0.6	1.4
TOTAL HYDROCARBONS	7.1	ND	3.8	8.0	8.5	4.9	8.7	11.9	6.3	8.6

TB/60 = Tubing bomb, 60 minutes
CONT = Continuous system

TABLE 15

PARTIAL CHROMATOGRAPHIC ANALYSES OF
NAPHTHA BEFORE/AFTER
AQUEOUS ACID/ALKALI STRIPPING*

COMPONENT	% OF TOTAL PEAK AREA	
	H31 NAPHTHA	ACID-ALKALI EXTRACTED H31 NAPHTHA
cyclohexane	0.8	0.8
pyridine	0.91	<0.1
benzene	5.6	5.6
toluene	2.4	2.5
o,m,p-xylene	11.2	12.7
methyl, ethylbenzene	5.4	5.5
phenol	0.83	<0.1
indan	14.1	14.3
cis decalin	10.2	10.4
m,p cresol	2.4	<0.1
trans decalin	2.9	3.1
tetralin	0.8	1.1
total identified	57.5	56.0
liquid-density (g/mL, 22°C)	0.796	0.790

*Stripping by 3 stages each of acid (0.1N H_2SO_4) and alkali (0.1N NaOH) followed by water wash and drying over Na_2SO_4.

TABLE 16

ELEMENTAL ANALYSES OF H31 NAPHTHA
BEFORE/AFTER AQUEOUS STRIPPING*

ELEMENT	ANALYSIS (%)	
	H31 Naphtha (C_5-190°C)	Extracted Naphtha
C	90.10	90.31
H	8.91	9.18
N	0.21	<0.05
S	0.08	<0.02
O	0.71	0.51

*Stripping by 3 stages each of acid (0.1N H_2SO_4) and alkali (0.1N NaOH) followed by water wash and drying over Na_2SO_4.

TABLE 17

RESULTS OF SEALED TUBE
THF STABILITY TESTS*

TEST SAMPLE	THF BENZENE GC PEAK AREA RATIO		INDICATED 96 h THF LOSS
	BEFORE	AFTER	(%)
FeXC Trench A Coal	1.0374	1.0275	0.95
Stainless Steel/Water	1.0620	0.9906	5.7
	1.0610	1.0006	5.2
Liquefaction Residues	1.0594	1.0536	0.58
	1.0536	1.0543	0
Control	1.0686	1.0639	0.44
	1.0652	1.0611	0.38

*Swagelok sealed 316 stainless steel tubes containing 1:1 THF-benzene mixture and test substrates; sealed samples held 96 h at 105°C

TABLE 18

COMPARISON OF UNIT OPERATIONS
FOR INDIRECT VS DIRECT LIQUEFACTION

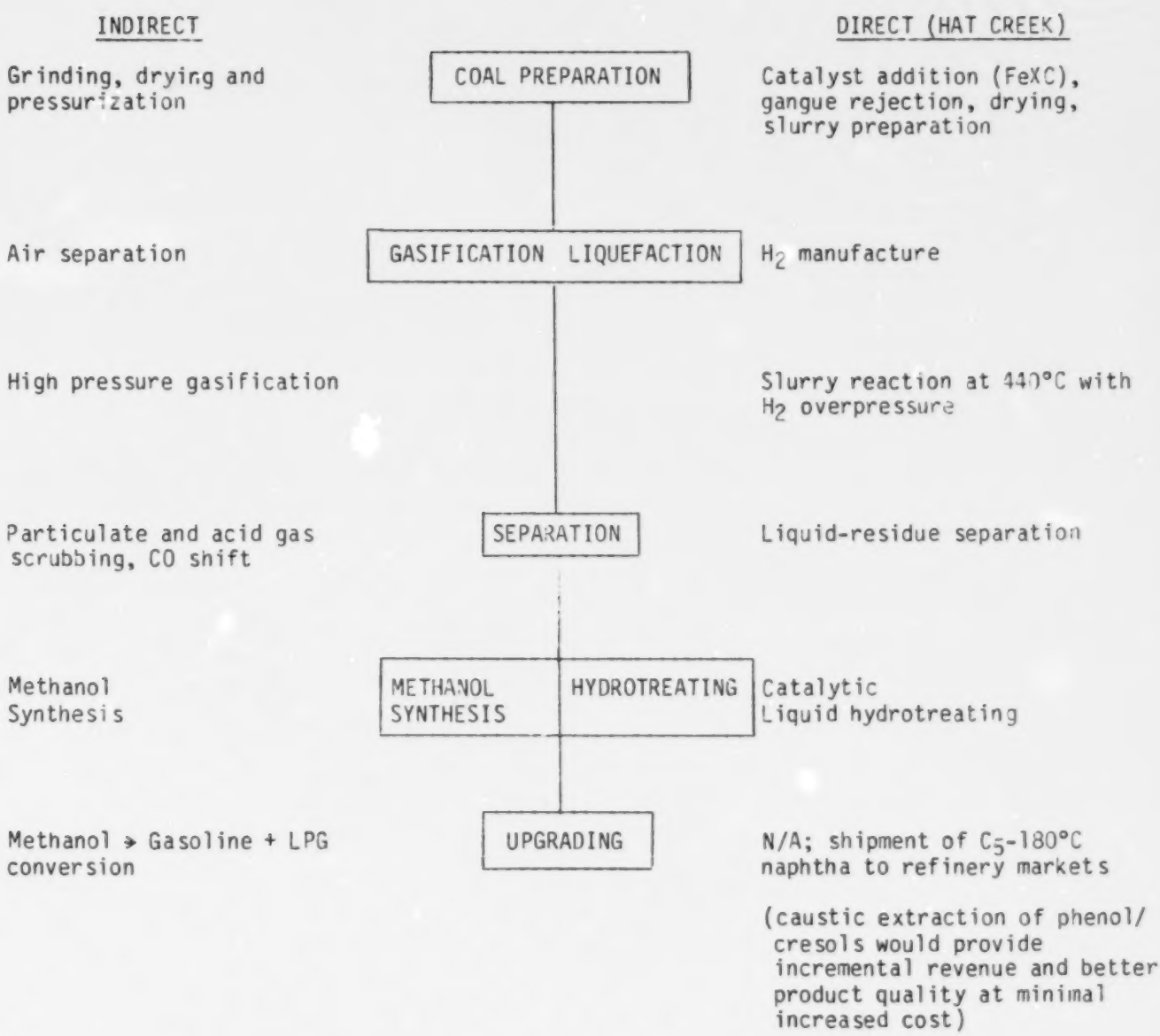


TABLE 19

COMPARISON OF COST AND REVENUE ESTIMATES FOR DIRECT LIQUEFACTION OF HAT CREEK COAL VS. INDIRECT LIQUEFACTION OF EAST KOOTENAY COAL WASTES

	INDIRECT LIQUEFACTION AT SPARWOOD	DIRECT LIQUEFACTION AT HAT CREEK
Capital Investment	$1.42 x 10^9 ($73,800/nominal daily bbl)	$1.2 x 10^9 ($60,000/nominal daily bbl)
Manufacturing Costs (Annual) Coal	$19 x 10^6 (9600 mtpd @ $6/t)	$49.5 x 10^6 (10,000 mtpd @ $15/t)
Power ($0.02/kwh)	$39 x 10^6	$10 x 10^6
Catalysts & Royalties	$35 x 10^6	$35 x 10^6
Fixed Costs (700 operating staff, maintenance, local taxes, etc.)	$81 x 10^6	$81 x 10^6
Total	$174 x 10^6/year	$175.5 x 10^6/year
Products	Mogas 16,000 bbl/d LPG 3,750 bbl/d	Naphtha 20,000 bbl/d (C_5 - 190°C)
Gross Revenue (330 operating days/year)	Mogas $251 x 10^6/year ($47.60/bbl or $0.30/L incl. tax remission subsidy) LPG $28 x 10^6/year	Naphtha $264 x 10^6/year ($40/bbl)
Total	$279 x 10^6/year (includes tax remission)	$264 x 10^6/year ($302x10^6/year with tax remission)
Net Revenue	$105 x 10^6/year (7.3% of capital investment)	$88.5 x 10^6/year (7.4% of capital investment) or $124.5x$10^6$/year (10.4% of capital investment) with tax remission
Discount Cash Flow Rate of Return	17.5%	

FIGURE 1

SCHEMATIC DIAGRAM OF TUBING BOMB REACTOR

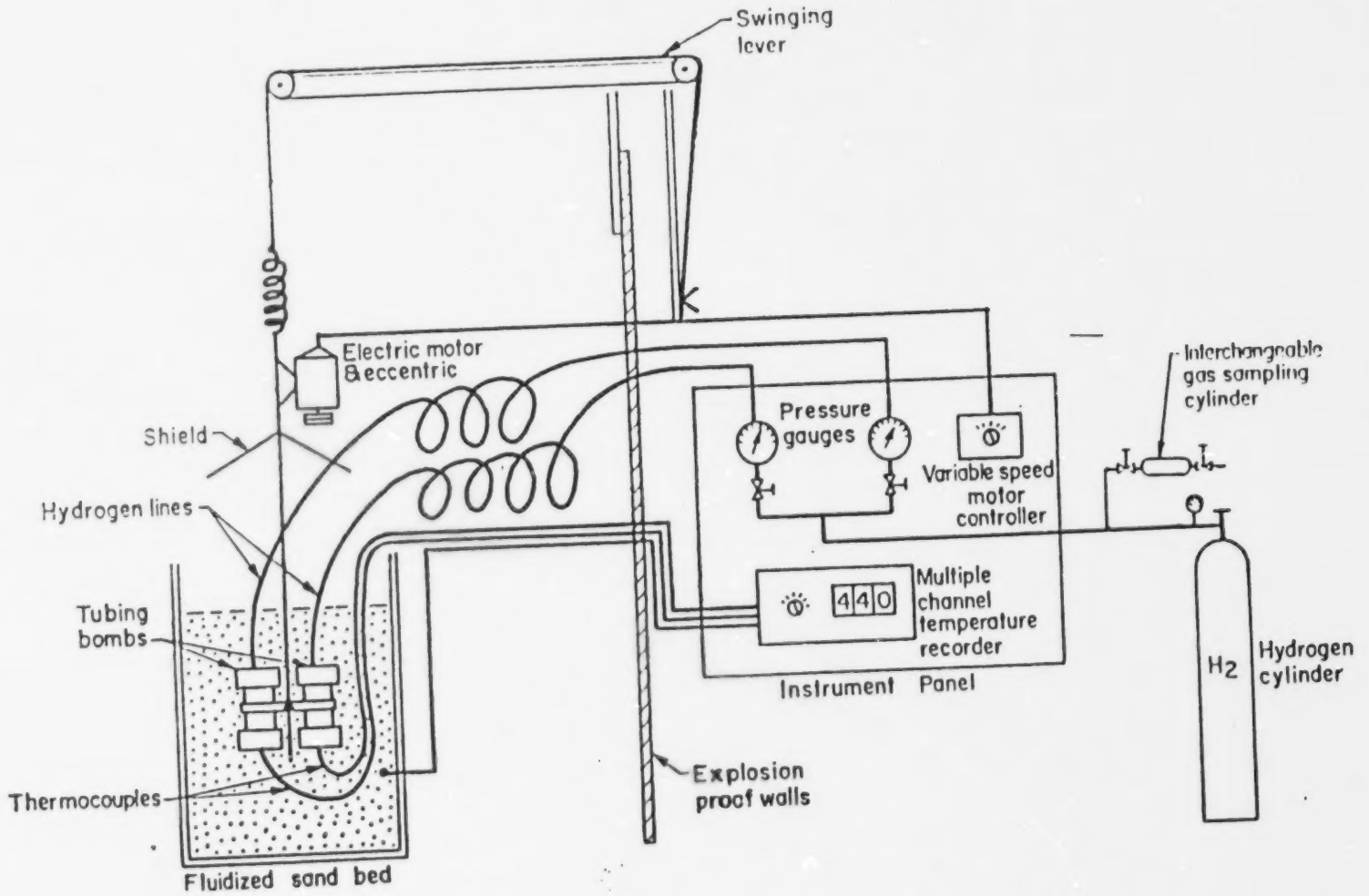


FIGURE 2: CONTINUOUS COAL LIQUEFACTION UNIT

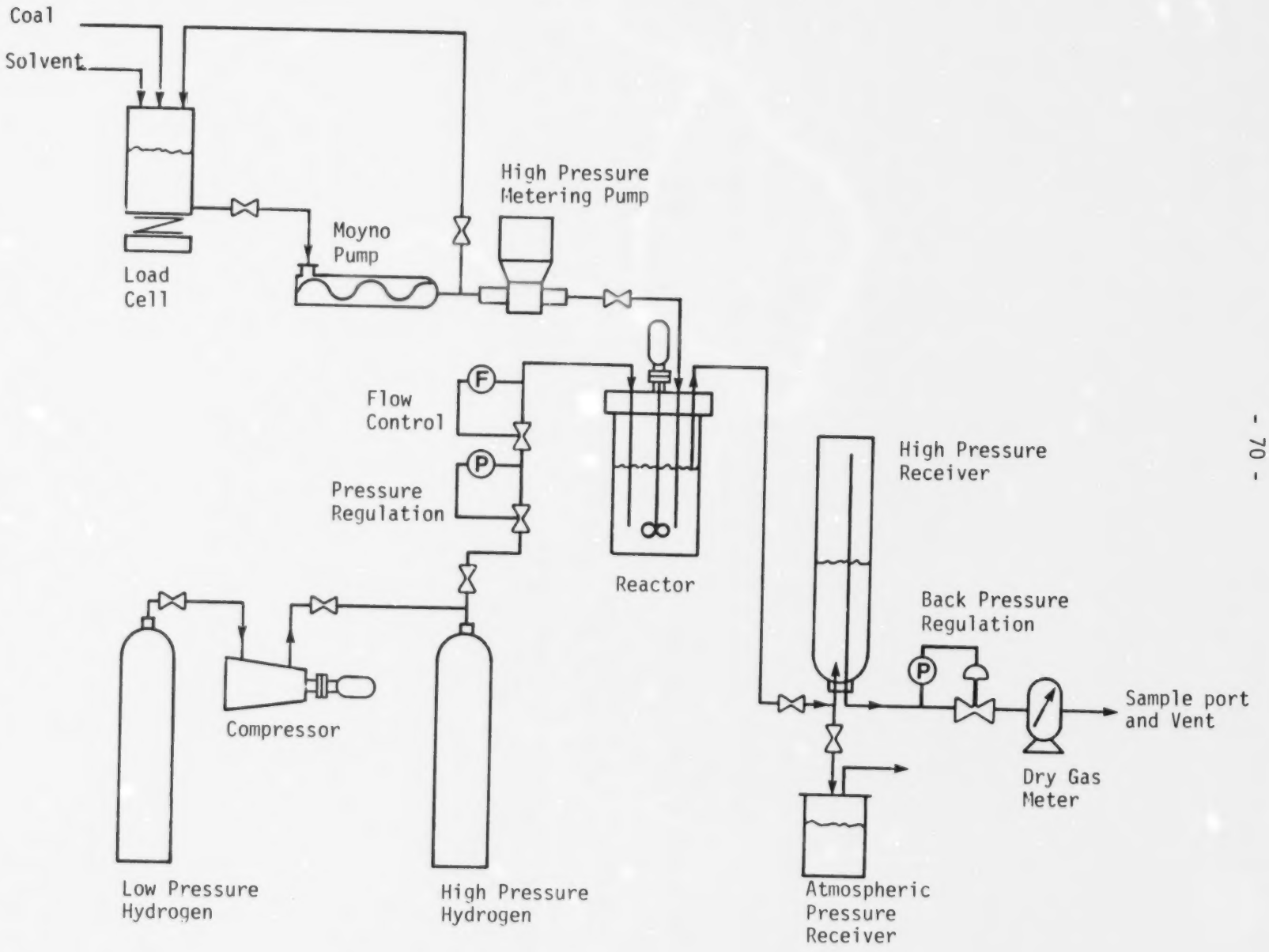


FIGURE 3: CONTINUOUS HYDROTREATER

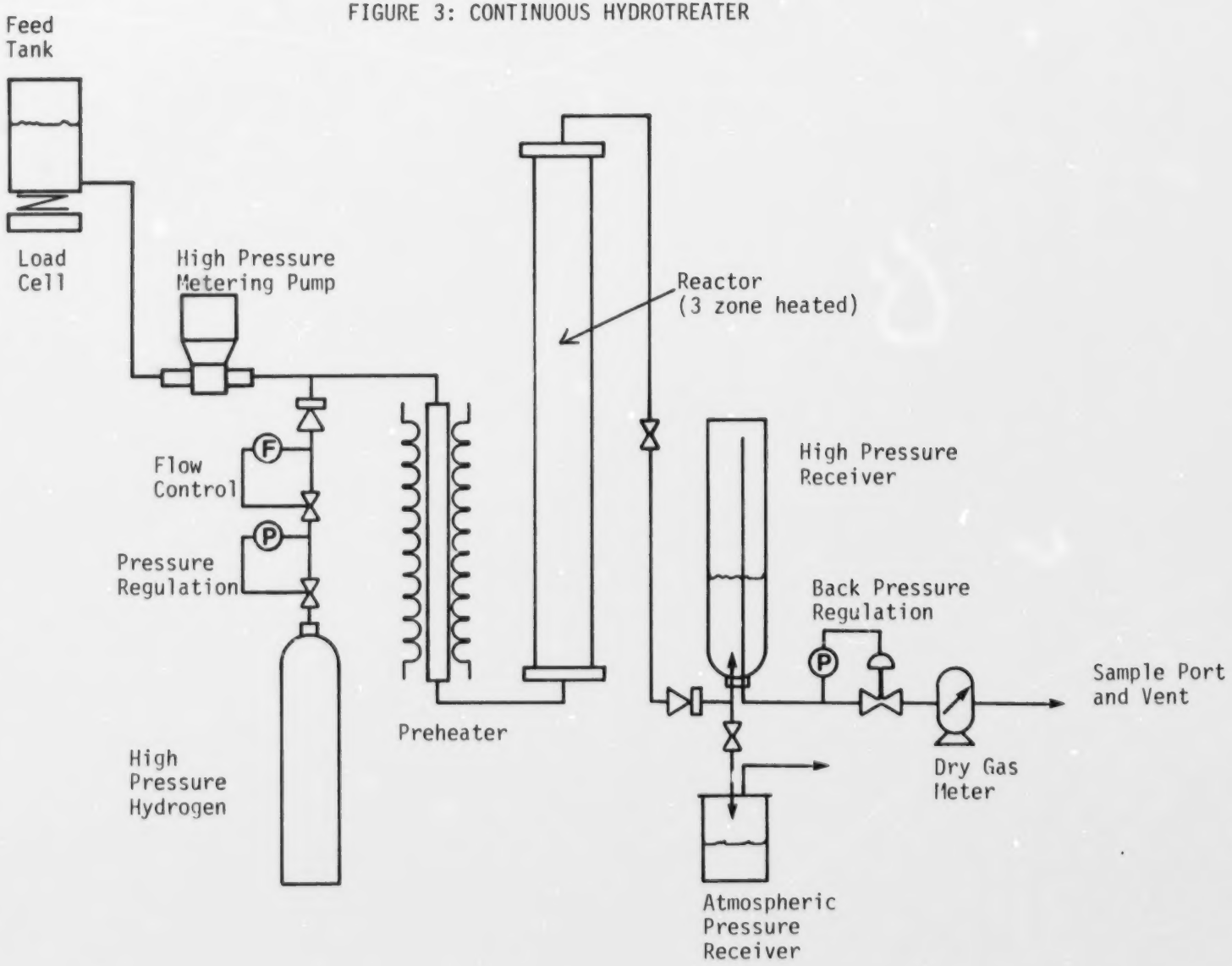


Figure 4: Thermogravimetric Analysis of C37 Primary Liquid Product

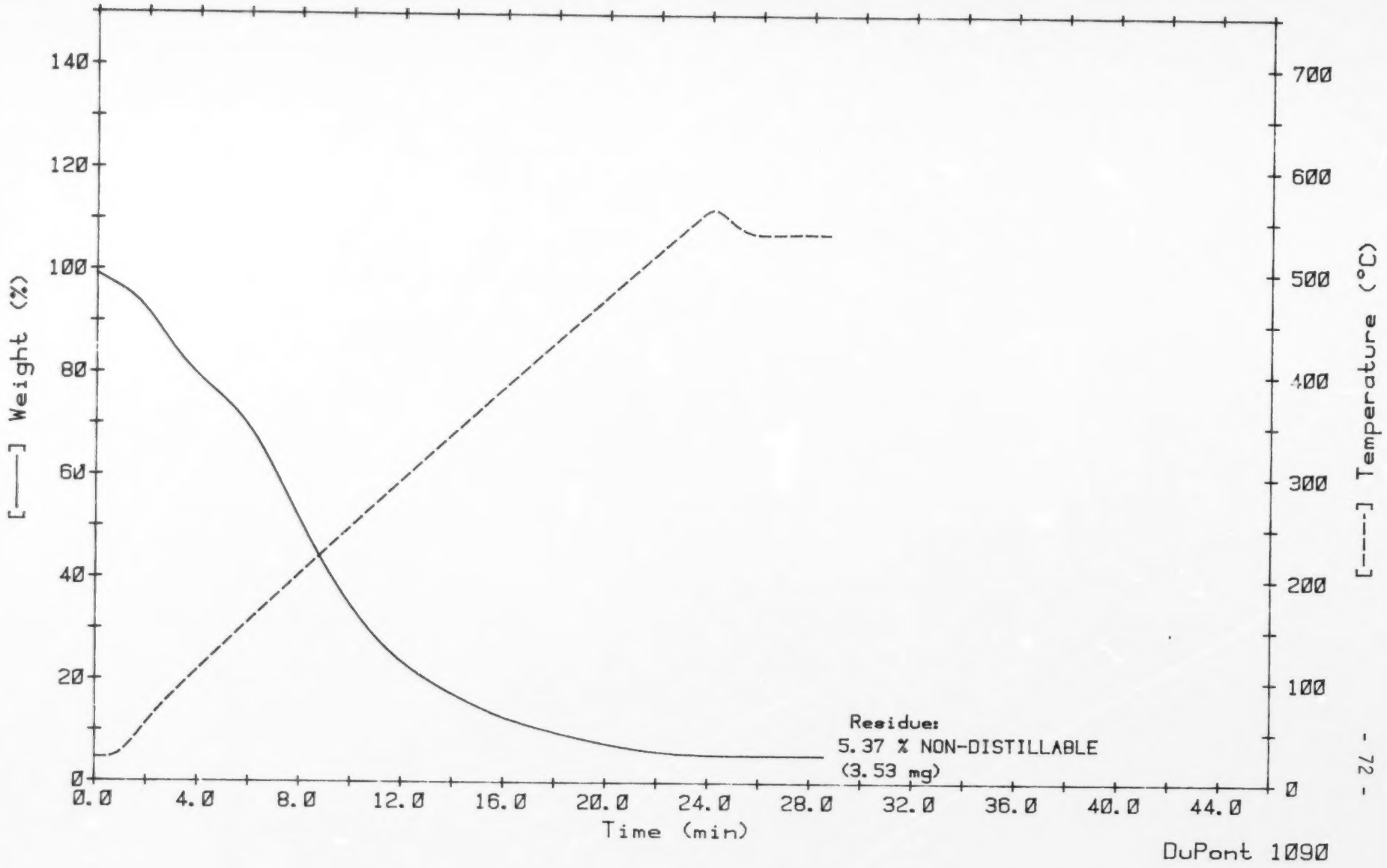


Figure 5: Thermogravimetric Analysis of C37 Sludge Product

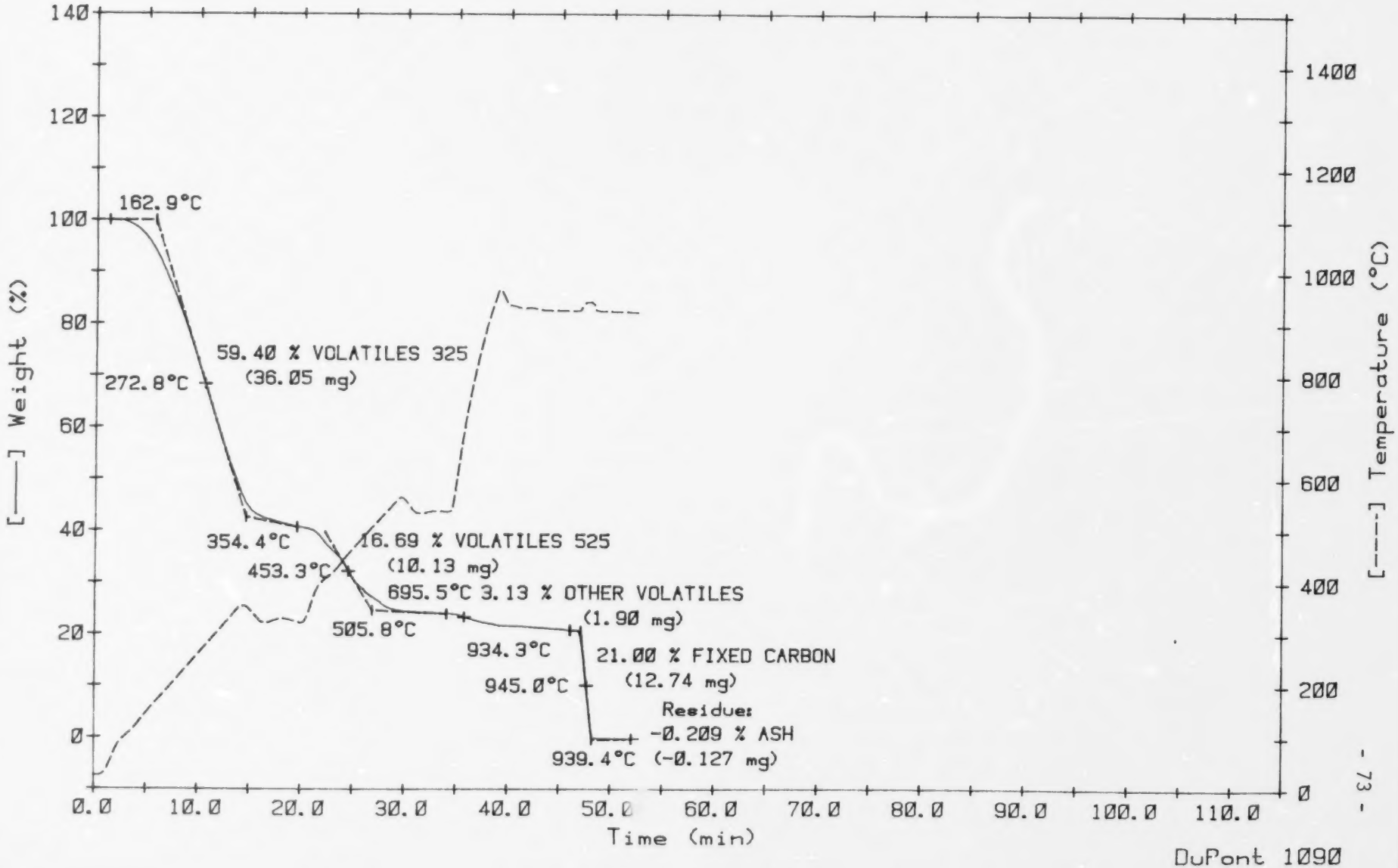


Figure 6: Thermogravimetric Analysis of C37 Product (+190°C) After Hydrotreating

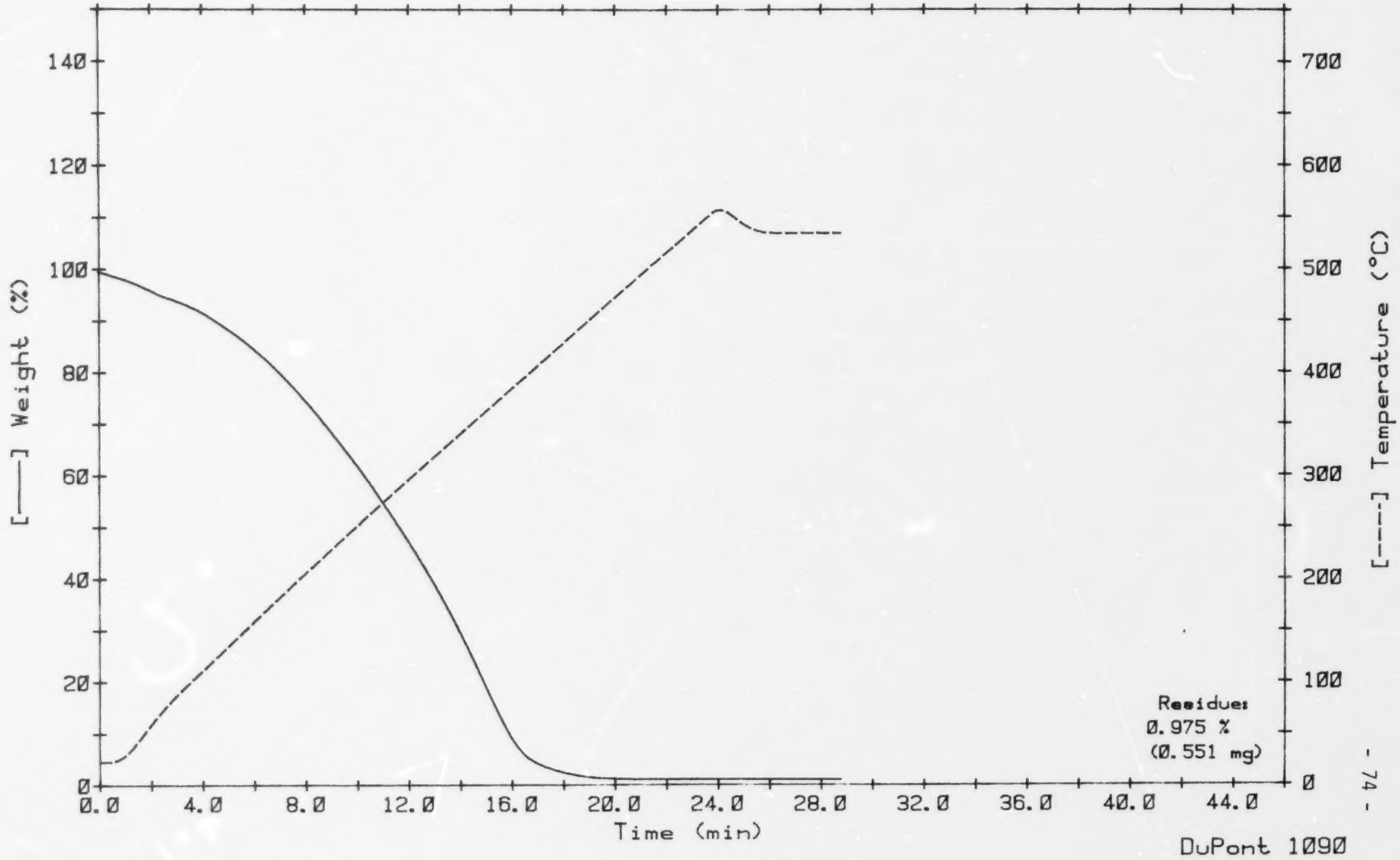
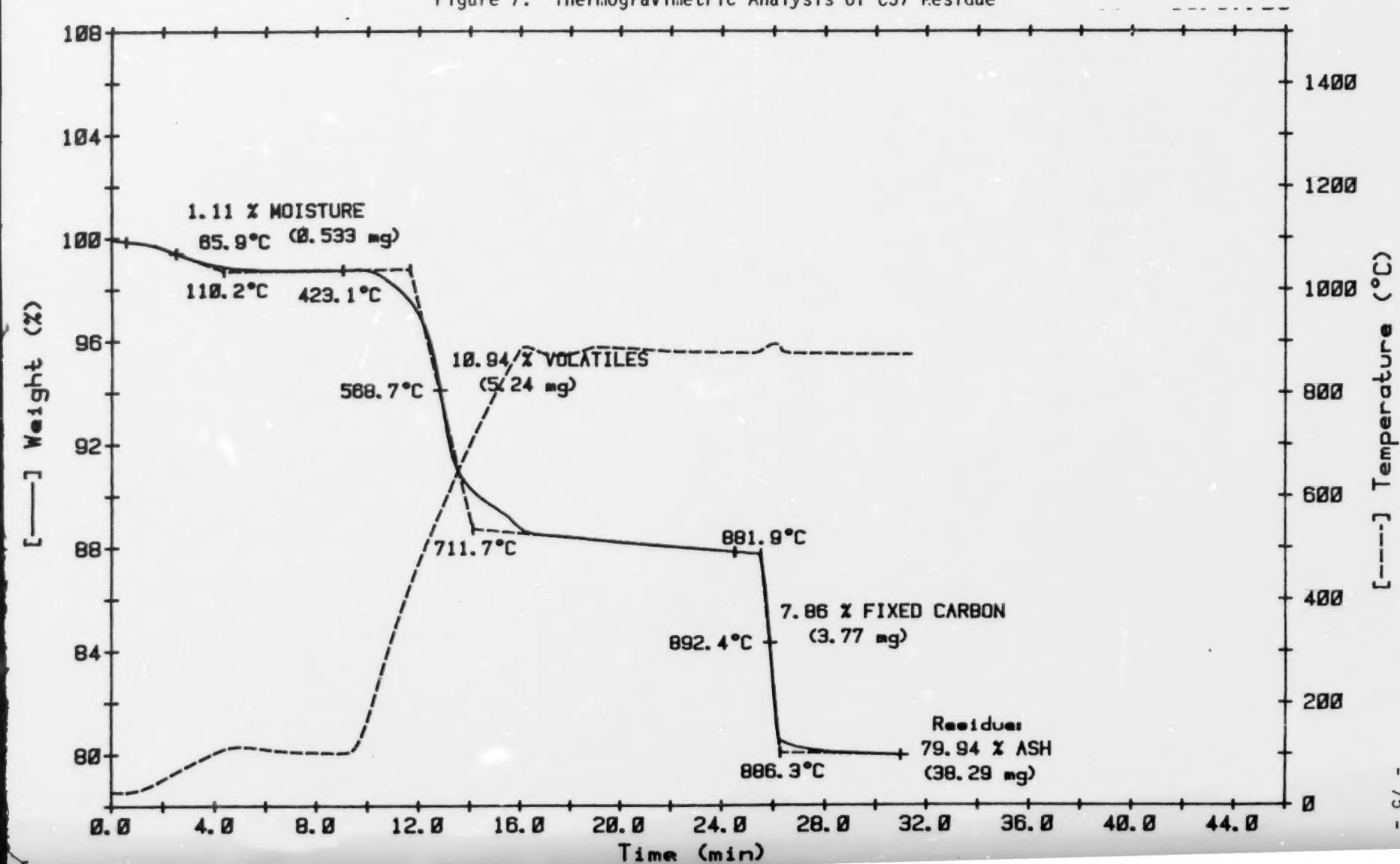


Figure 7: Thermogravimetric Analysis of C37 Residue



APPENDIX 1

SUMMARIES OF HYDROTREATER OPERATIONAL TEST DATA

HYDROTREATER RUN H17 SUMMARY

Feed Material: Raw Creosote

Conditions of Equilibrium:

Temperature	: 310	°C
Pressure	: 10.3	MPa
Liquid Feedrate:	1.12	kg/hr
H_2 Feed Rate	: 2.5	sL/min

Duration: 4.00 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	4480 g	Liquid	3360* g
Hydrogen	53 g	Hydrogen	36 g
		Gas	17 g
TOTAL	4533 g	TOTAL	3713 g

81.9 % of Material IN

Gas Production: 0.4 g/100 g of Liquid Feed

Hydrogen Consumption: 0.4 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	180°-200°C	+200°C
Yield** (% of liquid product)	18	16	66

Remarks:

* Low liquid recovery. Apparatus stripped down before run H17. All lines and reactor empty. Reactor not drained after the run.

**Distillation without reflux head.

HYDROTREATER RUN H18 SUMMARY

Feed Material: Raw Creosote

Conditions of Equilibrium:

Temperature	: 325	°C
Pressure	: 10.3	MPa
Liquid Feedrate:	1.21	kg/hr
H_2 Feed Rate	: 2.5	sL/min

Duration: 5.00 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	6050 g	Liquid	6103 g
Hydrogen	67 g	Hydrogen	55 g
TOTAL	6117 g	Gas	25 g
		TOTAL	6183 g

101.1 % of Material IN

Gas Production: 0.4 g/100 g of Liquid Feed

Hydrogen Consumption: 0.2 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	180°-200°C	+200°C
Yield* (% of Liquid product)	20	11	69

Remarks:

* Distillation without reflux head.

HYDROTREATER RUN H19 SUMMARY

Feed Material: Raw Creosote

Conditions of Equilibrium:

Temperature	:	325 °C
Pressure	:	13.8 MPa
Liquid Feedrate:		1.16 kg/hr
H_2 Feed Rate	:	5.0 sL/min

Duration: 6.52 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	7563 g	Liquid	7309 g
Hydrogen	174 g	Hydrogen	118 g
		Gas	29 g
TOTAL	7737 g	TOTAL	7456 g

96.4 % of Material IN

Gas Production: 0.4 g/100 g of Liquid Feed

Hydrogen Consumption: 0.7 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	180°-200°C	+200°C
Yield* (% of liquid product)	15	17	68

Remarks:

* Distillation without reflux head.

HYDROTREATER RUN H2O SUMMARY

Feed Material: Raw Creosote

Conditions of Equilibrium:

Temperature	: 325	°C
Pressure	: 13.8	MPa
Liquid Feedrate:	1.14	kg/hr
H_2 Feed Rate	: 2.5	sL/min

Duration: 6.00 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	6814 g	Liquid	6848 g
Hydrogen	80 g	Hydrogen	58 g
TOTAL	6894 g	Gas	230 g
		TOTAL	7136 g

103.5 % of Material IN

Gas Production: 3.4* g/100 g of Liquid Feed

Hydrogen Consumption: 0.3 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	180°-200°C	+200°C
Yield** (% of liquid product)	16	14	70

Remarks:

* High gas value probably due to problems in gas sampling.

**Distillation without reflux head.

HYDROTREATER RUN H21 SUMMARY

Feed Material: Raw Creosote

Conditions of Equilibrium:

Temperature	: 325	°C
Pressure	: 13.8	MPa
Liquid Feedrate:	1.80	kg/hr
H_2 Feed Rate	: 5.0	sL/min

Duration: 6.17 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	11130 g	Liquid	10846 g
Hydrogen	165 g	Hydrogen	161 g
		Gas	27 g
TOTAL	11295 g	TOTAL	11034 g

97.7 % of Material IN

Gas Production: 0.2 g/100 g of Liquid Feed

Hydrogen Consumption: 0.04 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	175°-185°C	185°C-200°C	+200°C
Yield* (% of liquid product)	13	11	7	69

Remarks:

* Distillation without reflux head.

HYDROTREATER RUN H22 SUMMARY

Feed Material: Raw Creosote

Conditions of Equilibrium:

Temperature	: 325	°C
Pressure	: 13.8	MPa
Liquid Feedrate:	1.73	kg/hr
H_2 Feed Rate	: 5.0	sL/min

Duration: 5.50 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	9525 g	Liquid	9968 g
Hydrogen	147 g	Hydrogen	114 g
TOTAL	9672 g	Gas	11 g
		TOTAL	10093 g

104.3 % of Material IN

Gas Production: 0.1 g/100 g of Liquid Feed

Hydrogen Consumption: 0.3 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	180°-200°C	+200°C
Yield* (% of liquid product)	20	7	73

Remarks:

*Distillation without reflux head.

HYDROTREATER RUN H23 SUMMARY

Feed Material: Raw Creosote

Conditions of Equilibrium:

Temperature	: 325	°C
Pressure	: 13.8	MPa
Liquid Feedrate:	1.77	kg/hr
H_2 Feed Rate	: 5.0	sL/min

Duration: 5.87 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	10374 g	Liquid*	10018 g
Hydrogen	157 g	Hydrogen	122 g
		Gas	21 g
TOTAL	9672 g	TOTAL	10161 g

96.5*% of Material IN

Gas Production: 0.2 g/100 g of Liquid Feed

Hydrogen Consumption: 0.3 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	175°-185°C	185°C-200°C	+200°C
Yield** (% of liquid product)	17	7	9	67

Remarks:

* Further 783 g of liquid obtained by draining the reactor. With this liquid the overall liquid recovery for runs H17 to H23 is 99.3%.

**Distillation without reflux head.

HYDROTREATER RUN H24 SUMMARY

Feed Material: Raw Creosote

Conditions of Equilibrium:

Temperature	: 325	°C
Pressure	: 10.3	MPa
Liquid Feedrate:	1.77	kg/hr
H_2 Feed Rate	: 5.0	sL/min

Duration: 5.00 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	8856 g	Liquid*	8839 g
Hydrogen	134 g	Hydrogen	104 g
TOTAL	8990 g	Gas	77 g
		TOTAL	9013 g

100.3 % of Material IN

Gas Production: 0.9 g/100 g of Liquid Feed

Hydrogen Consumption: 0.3 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	180°-200°C	+200°C
Yield** (% of liquid product)	18	7	75

Remarks:

*Includes liquid drained from the reactor after the run.

**Distillation without reflux head.

HYDROTREATER RUN H25 SUMMARY

Feed Material: Mixed +200°C fractions of H17-H24 products

Conditions of Equilibrium:

Temperature	: 325	°C
Pressure	: 13.8	MPa
Liquid Feedrate:	2.20	kg/hr
H_2 Feed Rate	: 5.0	sL/min

Duration: 4.47 hrs

MATERIAL BALANCE (H25 + H26)

Material IN		Material OUT	
Liquid	9828 g	Liquid**	9432 g
Hydrogen	120 g	Hydrogen	112 g
TOTAL	1948 g	Gas	65 g
		TOTAL	9606 g

96.6 % of Material IN

Gas Production: 0.7 g/100 g of Liquid Feed

Hydrogen Consumption: 0.1 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 195°C	+200°C
Yield (% of liquid product)	16	84

Remarks:

* Average feedrate. Feedrate changed during the run.
**Fraction not drained.
* Distillation without reflux head.

HYDROTREATER RUN H26 SUMMARY

Feed Material: Mixed +200°C fractions of H17-H24 products

Conditions of Equilibrium:

Temperature	:	325 °C
Pressure	:	13.8 MPa
Liquid Feedrate	:	2.17 kg/hr
H_2 Feed Rate	:	5.0 sL/min

Duration: 5.40 hrs

MATERIAL BALANCE (H25 + H26)

Material IN		Material OUT	
Liquid	11694 g	Liquid*	12156 g
Hydrogen	145 g	Hydrogen	125 g
		Gas	185 g
TOTAL	1948 g	TOTAL	12466 g

105.3 % of Material IN

Gas Production: 1.6 g/100 g of Liquid Feed

Hydrogen Consumption: 1.07 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 195°C	+200°C
Yield (% of liquid product)	21	79

Remarks:

* Includes 342 g of liquid drained from the reactor. Material balance over runs H25 and H26 yields 101.3%.
**Gas yield inflated due to problems in sampling.
* Distillation without reflux head.

HYDROTREATER RUN H27 SUMMARY

Feed Material: C30 + C34 liquid product

Conditions of Equilibrium:

Temperature	:	350 °C
Pressure	:	13.8 MPa
Liquid Feedrate:		1.98 kg/hr
H_2 Feed Rate	:	5.02 sL/min

Duration: 4.53 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	8795 g	Liquid	9012 g
Hydrogen	122 g	Hydrogen	102 g
		Gas	19 g
TOTAL	9097 g	TOTAL	9133 g

100.4% of Material IN

Gas Production: 0.22 g/100 g of Liquid Feed

Hydrogen Consumption: 0.2 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	+180°
Yield (% of liquid product)	14	86

Remarks:

HYDROTREATER RUN H28 SUMMARY

Feed Material: C32 + C33 liquid product

Conditions of Equilibrium:

Temperature	: 350	°C
Pressure	: 13.8	MPa
Liquid Feedrate:	1.97	kg/hr
H_2 Feed Rate	: 5.0	sL/min

Duration: 3.27 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	6842 g	Liquid	6791 g
Hydrogen	88 g	Hydrogen	80 g
		Gas	19 g
TOTAL	6920 g	TOTAL	6890 g

99.6% of Material IN

Gas Production: 0.28 g/100 g of Liquid Feed

Hydrogen Consumption: 0.1 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	+180°
Yield (% of liquid product)	8	92

Remarks:

HYDROTREATER RUN H29 SUMMARY

Feed Material: C35 liquid product

Conditions of Equilibrium:

Temperature	:	350 °C
Pressure	:	13.8 MPa
Liquid Feedrate:		1.99 kg/hr
H_2 Feed Rate	:	5.02 sL/min

Duration: 3.65 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	7279 g	Liquid	7240 g
Hydrogen	98 g	Hydrogen	93 g
TOTAL	7377 g	Gas	21 g
		TOTAL	7354 g

99.7% of Material IN

Gas Production: 0.29 g/100 g of Liquid Feed

Hydrogen Consumption: 0.1 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	+180°
Yield (% of liquid product)	9	91

Remarks:

HYDROTREATER RUN H30 SUMMARY

Feed Material: C36 liquid product

Conditions of Equilibrium:

Temperature	:	350 °C
Pressure	:	13.8 MPa
Liquid Feedback	:	2.09 kg/hr
H_2 Feed Rate	:	5.03 sl/min

Duration: 2.70 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	5656 g	Liquid	5777 g
Hydrogen	73 g	Hydrogen	72 g
TOTAL	5729 g	Gas	20 g
		TOTAL	5869 g

102.4% of Material IN

Gas Production: 0.35 g/100 g of Liquid Feed

Hydrogen Consumption: 0.02 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	+180°
Yield (% of liquid product)	10	90

Remarks:

HYDROTREATER RUN H31 SUMMARY

Feed Material: C37 liquid product

Conditions of Equilibrium:

Temperature	:	350 °C
Pressure	:	13.8 MPa
Liquid Feedrate:		1.99 kg/hr
H_2 Feed Rate	:	5.06 sL/min

Duration: 2.08 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	4154 g	Liquid	4119 g
Hydrogen	56 g	Hydrogen	56 g
TOTAL	4210 g	Gas	4 g
		TOTAL	4179 g

99.2% of Material IN

Gas Production: 0.1 g/100 g of Liquid Feed

Hydrogen Consumption: - g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	+180°
Yield (% of liquid product)		

Remarks:

HYDROTREATER RUN H32 SUMMARY

Feed Material: Raw Creosote

Conditions of Equilibrium:

Temperature	: 325	°C
Pressure	: 13.8	MPa
Liquid Feedrate:	1.07	kg/hr
H_2 Feed Rate	: 5.06	sL/min

Duration: 5.58 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Liquid	5964 g	Liquid	6040 g
Hydrogen	151 g	Hydrogen	108 g
TOTAL	6115 g	Gas	6 g
		TOTAL	6154 g

100.6% of Material IN

Gas Production: 0.1 g/100 g of Liquid Feed

Hydrogen Consumption: 0.7 g/100 g of Liquid Feed

DISTILLATION

Fraction:	i.b.p. - 180°C	+180°
Yield (% of liquid product)		

Remarks:

APPENDIX 2

SUMMARIES OF CONTINUOUS LIQUEFACTION OPERATIONAL TEST DATA

CONTINUOUS LIQUEFACTION RUN C33 SUMMARY

Feed Material:
COAL: HAT CREEK Trench A C-04-2
LIQUID: Mixed +200°C fractions of hydrotreated creosote (H17-H24)
SLURRY DENSITY: 26.2% (13.0% daf)

Conditions of Equilibrium:

Temperature	:	440 °C
Pressure	:	13.8 MPa
Liquid Feedrate:		1.44 kg/hr
H_2 Feed Rate	:	5.0 sL/min

Initial Reactor Load: 1,136 g
Duration: 5.50 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Slurry	8995 g	Slurry	8383 g
H_2	147 g	H_2*	118 g
TOTAL	9142 g	Gas*	245 g
		TOTAL	8746 g

95.7% of Material IN

Gas Production:* 21% daf
Hydrogen Consumption:* 0.8% daf

LIQUID-SOLID SEPARATION

Phase:	Liquid	Sludge	Solid**
Yield:	65.9	5.5	28.6
(% of product slurry)			(13.3)

Remarks:

* Estimated for this run from tubing bomb liquefaction results.
**Solids by difference.

CONTINUOUS LIQUEFACTION RUN C34 SUMMARY

Feed Material:*
COAL: HAT CREEK Trench B Fe-ex C-04-2
LIQUID: +200°C fractions of hydrotreated creosote (H25)
SLURRY DENSITY: 37.5% (25.01% daf)

Conditions of Equilibrium:

Temperature	:	440 °C
Pressure	:	13.8 MPa
Liquid Feedrate	:	1.37 kg/hr
H_2 Feed Rate	:	5.0 sL/min

Initial Reactor Load: 1,040 g
Duration: 5.50 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Slurry	8551 g	Slurry	8014 g
H_2	147 g	H_2*	69 g
TOTAL	8698 g	Gas*	408 g
		TOTAL	8491 g

97.6% of Material IN

Gas Production:* 19.1% daf
Hydrogen Consumption:* 3.6% daf

LIQUID-SOLID SEPARATION

Phase:	Liquid	Sludge	Solid**
Yield:	71.7	9.9	19.4
(% of product slurry)			(12.4)

Remarks:

* Approx. 1 g of CS_2 per 100 g of coal added to the slurry.
**Solids by difference.

CONTINUOUS LIQUEFACTION RUN C35 SUMMARY

Feed Material:*
COAL: HAT CREEK Trench B Fe-ex C-04-2
LIQUID: +180°C fractions of hydrotreated C30+C34 liquids (H27)
SLURRY DENSITY: 37.7% (26.0% daf)

Conditions of Equilibrium:

Temperature	:	440 °C
Pressure	:	13.8 MPa
Liquid Feedrate:		1.15 kg/hr
H_2 Feed Rate	:	5.0 sL/min

Initial Reactor Load: 1,175 g
Duration: 9.00 hrs

MATERIAL BALANCE

Material IN		Material OUT	
Slurry	11549 g	Slurry	10682 g
H_2	242 g	H_2	230 g
TOTAL	11791 g	Gas	562 g
		TOTAL	11474 g

97.3% of Material IN

Gas Production: 18.7% daf
Hydrogen Consumption: 0.4% daf

LIQUID-SOLID SEPARATION

Phase:	Liquid	Sludge	Solid**
Yield: (% of product slurry)	75.6	12.3	12.1

Remarks:

* Approx. 10 g of CS_2 per 1 kg of coal added to the slurry.
**Solids by difference.

CONTINUOUS LIQUEFACTION RUN C36 SUMMARY

Feed Material:*
COAL: HAT CREEK Trench A Fe-ex C-03-2
LIQUID: +180°C fractions of hydrotreated C35 liquid (H29)
SLURRY DENSITY: 29.4% (18.6% daf)

Conditions of Equilibrium:

Temperature	:	440 °C
Pressure	:	13.8 MPa
Liquid Feedrate	:	1.24 kg/hr
H_2 Feed Rate	:	5.0 sL/min

Initial Reactor Load: 1,026 g
Duration: 6.50 hrs

MATERIAL BALANCE

Material IN		Material OUT**	
Slurry	9102 g	Slurry	8394 g
H_2	175 g	H_2	165 g
TOTAL	9277 g	Gas	420 g
		TOTAL	9022 g

96.8% of Material IN

Gas Production: 24.8% daf
Hydrogen Consumption: 0.6% daf

LIQUID-SOLID SEPARATION

Phase:	Liquid	Sludge	Solid+
Yield: (% of product slurry)	71.1	11.3	17.6

Remarks:

* Approx. 10 g of CS_2 per 1 kg of coal added to the slurry.
**Additional 450 g of material was recovered by flushing the receiver with THF. The overall material balance for runs C33 to C36 is 98.1%.
+ Solids by difference.

CONTINUOUS LIQUEFACTION RUN C37 SUMMARY

Feed Material:*
COAL: HAT CREEK Trench A Fe-ex C-03-2
LIQUID: +190°C fractions of hydrotreated C36 liquid (H30)
SLURRY DENSITY: 28.7% (19.1% daf)

Conditions of Equilibrium:

Temperature	:	440 °C
Pressure	:	13.8 MPa
Liquid Feedrate	:	1.25 kg/hr
H_2 Feed Rate	:	5.0 sL/min

Initial Reactor Load: 961 g
Duration: 5.62 hrs

MATERIAL BALANCE

Material IN		Material OUT**	
Slurry	7914 g	Slurry	7588 g
H_2	152 g	H_2	170 g
TOTAL	8067 g	Gas	224 g
		TOTAL	7982 g

98.9% of Material IN

Gas Production: 14.8% daf
Hydrogen Consumption:** N/O% daf

LIQUID-SOLID SEPARATION

Phase:	Liquid	Sludge	Solid+
Yield: (% of product slurry)	70	20	10

Remarks:

* Approx. 10 g of CS_2 per 1 kg of coal added to the slurry.
**Inflated H_2 probably due to sampling.
+ Some liquid and sludge lost during separation. Their yields were calculated by difference and by applying a proportional correction. As there was both proportionally more sludge than liquid, the sludge yield is a least reliable value.